Metallography of Tungsten

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TUNGSTEN has the highest melting point of all the known metals, namely 3350° C.; it is one of the hardest of the metals; it has the highest equiaxing or recrystallization temperature after strain hardening, of any pure metal known. It is particularly distinguished because, when composed of small equiaxed grains, it is extremely brittle and fragile at room temperature, and when possessing a fibrous structure it may be ductile and pliable at room temperature. The common ductile metals act in exactly the opposite manner in this respect.

The present paper will include a brief note regarding the manufacture of wrought and ductile tungsten. The metallography of wrought and ductile tungsten in the various stages of manufacture will be considered more or less in detail. The general relationships between the properties of tungsten and other metals will also be considered.

A discussion will be given explaining why fibrous tungsten is ductile at room temperature, even though past experience with other metals would indicate that it should be brittle. Finally, a brief note regarding some new fundamental metallographic propositions relating to all metals will be given.

MANUFACTURE OF WROUGHT AND DUCTILE TUNGSTEN

Up to about 10 years ago, it had not been possible to work tungsten mechanically. About the year 1908, Dr. William D. Coolidge, of the General Electric Co., produced ductile tungsten from what was then supposed to be an inherently brittle metal and which, in fact, was inherently brittle in its normal state, that is, when composed of equiaxed grains. Since that time many products, chief among which are the filaments for electric incandescent lamps, have been made from wrought and ductile tungsten.

Tungsten is produced chiefly from the minerals wolframite and scheelite.[1–8] The ores may be reduced in many ways. For example, the ore may be fused with alkali carbonates and the fusion dissolved in water; sodium tungstate is formed, which is soluble in water. This

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[1–8] References are to bibliography at end of paper.

may be changed to tungstic oxide, WO_3, by the addition of an acid to the sodium tungstate liquor. WO_3, a yellow precipitate, is separated by filtering. The tungsten oxide may then be purified to any desired degree by dissolving it in ammonia and then reprecipitating by the addition of acid, followed by filtering and washing.

For the manufacture of most of the wrought tungsten products, the tungsten oxide is treated as follows: after drying, sufficient thorium nitrate solution is added to make 0.75 per cent. thoria (ThO_2) in the oxide. The tungsten oxide is made into a batter with the thorium nitrate solution, additional water being added if necessary, after which it is thoroughly mixed. The mixture is then dried and placed in either a fire-clay or a silica crucible and heated to about 1100° C. for a period usually in excess of 1 hr. This is called the firing operation, and it has for its purpose the agglomerating of the extremely fine particles of tungsten oxide into larger, or coarser-grained particles. When the firing is done in a fire-clay crucible, some of the crucible material is dissolved by the oxide, and produces a beneficial effect when the metal is to be used for incandescent lamp filaments.

The fired oxide is then reduced by hydrogen to tungsten metal powder, at a temperature in the neighborhood of 1000° C. The tungsten powder thus produced is relatively coarse grained when compared to the tungsten powder from which the sintered tungsten filaments were made before the advent of drawn tungsten wire.

The tungsten powder is pressed dry, into any desirable shape or size. A common size and shape consists of a rod about 16 in. (40 cm.) long having a cross-section about 3/8 in. (9.5 mm.) square. The pressure on such a rod must be applied from the side of the mold and not from the end, else the resulting tungsten ingot will not be workable. The pressed tungsten slug is so fragile that it can be moved only by sliding it along a surface. It is moved in this manner onto a platform of tungsten, nickel, or other suitable material having a high melting point, and while still on the platform is inserted into an electric furnace of the tube type, which is heated to about 1200° or 1300° C., in which an atmosphere of hydrogen is maintained. This is called the baking operation. After baking, the tungsten slug may be handled without danger of breaking. At this stage, it is porous, containing about 40 per cent. voids.

The baked tungsten slug is then clamped into two electrodes, arranged in a vertical position, the bottom one of which floats in a bath of mercury cooled by a water jacket. This arrangement is necessary, because in the operations which follow, the tungsten slug shrinks, usually about 15 per cent. in length, and the mercury well must accommodate this shrinkage and still maintain electrical contact with the lower electrode. After the slug is securely clamped in the electrodes, a gas-tight housing is lowered around it and a current of hydrogen is passed through this housing. Electric current is then passed through the slug, thus heating it to an extremely high temperature. For most purposes, a temperature of about 3000° to 3200° C. is used, and this temperature is maintained for 10 or 15 min. This may be called the sintering operation.

The sintered tungsten ingot is substantially free from voids, having a density of about 18. The two ends which were clamped in the electrodes will not have been heated to a sufficiently high temperature to cause sintering, so they are broken off and then the ingot is ready for the mechanical working process.

The working operation is begun in swaging machines,[10–16] which operate the swaging dies. A pair of swaging dies consists of two similar blocks of high-speed steel, each block containing half the desired opening. The openings are circular in cross-section. The side of the steel block containing half the desired opening is called its "face." These two half dies are placed in the swaging machine face to face, sufficiently loose so that the swaging blow can be struck, when the die halves move away from and toward each other. The machine head rotates and the dies rotate with it. Centrifugal force moves the die halves apart and rollers in the head of the machine push them together again. The dies move backward and forward several thousand times per minute. The faces of these dies must be made in a special manner for the swaging of tungsten. The portions which come in contact with the metal to be worked must be very much shorter than when swaging the ordinary metals. These are called short-faced dies. They are made of high-speed steel because they are to be used to swage hot metal.

For beginning the swaging operation on a tungsten ingot having a square or rectangular cross-section, a pair of swaging dies is selected which will just round off the corners of the ingot. The ingot is put into an electric furnace, usually consisting of an alundum tube wound with tungsten or molybdenum wire, in which an atmosphere of hydrogen is maintained. The temperature of this furnace may be as high as 1700° C. but it is more frequently in the neighborhood of 1600° C. When the ingot has attained the temperature of the furnace, one end is grasped with a pair of tongs and the other end is quickly inserted into the swaging machine. It is not possible to swage the whole length of the rod in this manner, so the ends of the rod are reversed and it is again heated, after which the unswaged end is inserted into the swaging machine. A pair of swaging dies having a somewhat smaller opening than the first pair of dies used is now put into the machine; the ingot is heated again and the swaging operations continued as before. Four or five dies are required to make a round rod from a square or rectangular ingot.

When the tungsten rod is sufficiently elongated to enable the end which first passes through the swaging machine to be gripped from the exit side, only one heating for each pair of swaging dies is necessary. As the degree of work increases on the rod, the temperature is gradually decreased. When the working temperature has been reduced to about 1300° C., gas may be used to heat the metal preparatory to swaging. The temperature may be decreased in steps, that is, there may be a certain temperature maintained for a half dozen or more dies and then the temperature lowered for the next half dozen dies. These conditions will depend on the properties desired in the swaged tungsten rod. The swaging operation may begin on tungsten ingots of any size, but a common size is ¼ to ⅜ in. square (6.35 to 9.5 mm.), and 12 to 14 in. long (30.48 to 35.56 cm.).

If it is desired to produce wrought tungsten rods more than 0.030 in. (0.76 mm.) diameter, all of the work is done by swaging. If, however, it is desired to produce tungsten wires smaller than 0.030 in. diameter, the swaging operation is resorted to only until a size of 0.030 in. or thereabouts is reached. The temperature of swaging will have been reduced as the size of the rod decreased so that the 0.030 in. rod would be swaged at a temperature of 1000° or 1100° C.

When the tungsten ingot is removed from the furnace previous to swaging, the temperature corresponds to a brilliant white heat. Tungsten oxide has not formed in the furnace because of the hydrogen atmosphere, but as soon as the ingot is removed from the hydrogen atmosphere, its temperature is such that tungsten oxide forms very rapidly, volatilizes and condenses as a white fume at some distance above the rod itself.

The tungsten ingots may also be reduced by rolling. High-speed rolls are employed and the ingot is heated to 1500° C. before each pass. Rolls are used for flat pieces and swaging machines for round sections.

The sintered tungsten ingot is as brittle as glass and extremely fragile. It is composed of equiaxed grains such as may be seen in Fig. 1, which represents the structure of a sintered tungsten ingot. When the swaging has been continued until the rectangular cross-section of the ingot has been changed to circular, the structure has been changed to that shown in Fig. 2. With still further swaging, when the tungsten rod has reached a diameter of 0.125 in. (3.2 mm.), its structure is changed to that shown in Fig. 3. At a size of 0.082 in. (2.08 mm.) its structure has changed to one corresponding to that shown in Fig. 4, while Fig. 5 represents the structure when the tungsten rod has been swaged to a diameter of 0.030 in. (0.76 mm.).

It will be seen from these micrographs that the equiaxed grains composing the sintered tungsten ingot have been progressively elongated during the swaging operations into fibers of metallic tungsten. The 0.030-in. swaged rod shown in Fig. 5 is ductile cold. It can be bent cold or it can be drawn cold.

While the tensile strength of the sintered tungsten ingot from which this 0.030-in. swaged tungsten rod was reduced was only 18,000 lb. per square inch, the tensile strength of the rod was 215,000 lb. per square inch.

The sintered tungsten ingot was absolutely brittle, but the 0.030-in. swaged tungsten rod has an elongation of about 4 per cent. in 2 in. and a reduction in area at the point of fracture of about 28 per cent.

When it is desired to produce wires smaller than 0.030 in. diameter, a hot drawing process is resorted to. Diamond dies are usually employed for this purpose. The lubrication used, both for the swaging operations on the smaller rods and in the drawing process, is aquadag. The wire to be drawn is first passed through an aquadag mixture which is in the form of a thin paste, and then through a gas flame or other heating medium which heats the wire to the desired drawing temperature and also bakes on it a coating of the lubricant. The dies vary in size by small steps; that is, the diameter of the opening in one die will be only about 6 per cent. greater than that in the next following die.

The temperature at the beginning of the drawing may be in the neighborhood of 1000° C. and when the wire is reduced to the size of about 0.005 in. (0.127 mm.) diameter, the temperature of drawing will have been reduced to a dull red heat. Fig. 6 is a micrograph of a drawn tungsten wire. All of the tungsten wires so produced, no matter what their size, are ductile cold. The increase in tensile strength is continuous, the smallest wires being the strongest.

TABLE 1.—*Tensile Strength of Tungsten*

Kind of Material	Diam. in Mils	Tensile Strength in Lb. per Sq. In.
Sintered tungsten ingot	200 by 250	18,000
Swaged rod	216.0	50,600
Swaged rod	125.0	107,000
Swaged rod	80.0	176,600
Swaged rod	26.0	215,000
Drawn wire	18.0	264,000
Drawn wire	7.23	340,000
Drawn wire	5.78	366,000
Drawn wire	5.50	378,000
Drawn wire	3.96	483,000
Drawn wire	1.14	590,000

Table 1 shows the increase in tensile strength of tungsten products with the increase in mechanical working. Tungsten wire 1.14 mils diam. (a mil is 0.001 in.) shows a reduction of area at the point of fracture of about 65 per cent. Coupled with great strength, drawn tungsten wire possesses the property of ductility in the cold to a very usable degree. The wire can be coiled on mandrels having diameters but slightly greater than that of the wire itself. It can be bent into the various shapes necessary for lamp making, and it can be handled in long lengths without danger of breaking.

Table 1 shows that the tensile strength of the 1.14-mil drawn wire is about 33 times as great as that of the sintered tungsten ingot. In ordinary metal-working processes, an increase of tensile strength of six times that of the original starting material is unusual.

General Relationships Between Tungsten and Other Metals

It should be noted from the preceding that tungsten possesses several properties different from those of the common ductile metals. For example, when the common ductile metals are worked cold or at elevated temperatures below their annealing temperatures, they are rendered more brittle at room temperature. Tungsten cannot be worked at room temperature when it is composed of equiaxed grains. When it is worked at elevated temperatures below its equiaxing temperature in such a manner as to deform the grains permanently, its ductility at room temperature increases.

When the ductile metals which have been made brittle by mechanical working at room temperature, or at elevated temperatures below their annealing temperatures, are annealed, ductility is restored. When tungsten which has been made ductile by mechanical working at an elevated temperature below its equiaxing temperature is heated above the equiaxing temperature, its ductility at room temperature is lost.

The ductile metals are in their most ductile condition when they are composed of small equiaxed grains. Tungsten is in its most brittle condition when it is composed of small equiaxed grains.

When the ductile metals are heated in such a manner that coarse-grained structures are produced, they become less malleable and ductile at room temperature, as a result of the coarse-grained structures. When tungsten, however, is heated in such a manner as to produce extremely coarse-grained structures, the resulting tungsten product possesses, to a slight degree, the ability to be deformed, especially by pressure at room temperature. The utility of this type of deformability in tungsten is limited. Let us suppose, for the sake of argument, that we have a single grain of tungsten, and that we proceed to work this grain mechanically at room temperature. It becomes harder and more brittle, due to the strain hardening, and our first thought would be to heat it above its equiaxing temperature, so that it would regain its malleability and ductility. Above the equiaxing temperature this strain-hardened grain would form many equiaxed grains. We would have produced in this manner a piece of tungsten metal composed of small equiaxed grains. This material would be absolutely brittle at room temperature and no further working at room temperature would be possible. This piece of tungsten could, however, be mechanically worked at high temperatures by the processes described above.

Molybdenum has some properties similar to those of tungsten, and others similar to those of the common ductile metals. It is in a more ductile condition at room temperature when it is composed of fine grains than when composed of coarse grains. In this respect it is similar to the common ductile metals. When its grains are deformed by mechanical working at temperatures above room temperature and below its equiaxing temperature, its ductility at room temperature increases. In this respect it more nearly resembles tungsten.

Tantalum[35] resembles the common ductile metals in all of its working properties. Tantalum must be very pure and especially free from hydrogen or nitrogen to be in a ductile condition. When it is melted in a vacuum, it is found to be one of the most malleable and ductile of metals. Fig. 7 is a micrograph of a cold-drawn tantalum wire which was mechanically worked at room temperature, without any annealing, from a fused globule of tantalum to a wire 0.007 in. (0.177 mm.) in diameter. This wire has not even yet reached its maximum limit of cold working. Fig. 8 shows a piece of the same wire which was heated in a vacuum to 1600° C. for 5 min. It will be noted that recrystallization has taken place. This annealed wire is much more ductile than the cold-worked wire, thus showing that in these respects tantalum is similar to the common ductile metals. Fig. 9 is a micrograph of a piece of the same tantalum wire heated for 1 min., in an atmosphere of hydrogen, to 1600° C. Recrystallization has taken place, but this wire has absorbed enough hydrogen to make it extremely brittle and fragile at room temperature. The properties of tungsten wire are the same whether heated in a vacuum or in hydrogen.

Cast zinc or worked zinc made coarse grained by a high-temperature annealing may be brittle at room temperature and workable at 150° C., and after this working the ductility at room temperature will be greater than before working. It is now known that this increase in ductility of zinc is due to a substitution of a fine-grained structure for the coarse-grained one. It is known that 150° C. is above the annealing temperature of worked zinc and consequently in the hot-working region. The refinement of the grain in zinc by working at 100° to 150° C. is analogous to the breaking down of the coarse grains in a steel ingot by working above a red heat.[36] No amount of work on tungsten above its annealing temperature will make it ductile at room temperature.

Platinum may be produced in a coherent malleable and ductile form by the Wolloston process[37] without resorting to the fusion operation. There are several points of difference between the Wolloston method for producing coherent ductile platinum without fusion and the method of producing coherent ductile tungsten without fusion. The platinum has only to be made coherent and it is inherently ductile. Tungsten has to be made coherent and then ductilized by the working process. The platinum cake immediately before working contains about 40 per cent. voids and these are welded by the working. The voids must be eliminated in tungsten before the working begins, else the material will not be workable.

Effect of Thoria on the Structure of Sintered Tungsten Ingots

The principal need for thoria or other non-metallic substances in tungsten which is to be used for lamp filaments is to control the direction, and, to a certain extent, the degree of grain growth in the filament, in such a manner as to render them both rugged (not easily broken by rough handling of the lamps) and free from off-setting.* With the old type of pressed tungsten filament, the purity of the tungsten did not have to be considered except for the functioning of the tungsten wire as a lamp filament. By the present method of manufacture of lamp filaments, however, the sintered tungsten ingots must be capable of being mechanically worked hot. Therefore the addition of thoria or other non-metallic substance must be made judiciously and the heat treatment of the tungsten ingot must also be varied to suit the thoria content. Tungsten containing more than 2 per cent. thoria is difficult to work into small wires.

The effect of the thoria is to increase the resistance to grain growth at high temperatures. Other non-metallic substances will offer resistance to grain growth in proportion to the volume, arrangement, and size of globules in the metal. It is difficult, however, to retain the more volatile oxides in the ingot. Even silica vaporizes almost completely during the sintering operation. Alumina volatilizes more slowly and thoria very little.

Plate 1 shows the variation in grain size of tungsten ingot with variation on the quantity of thoria, the temperature being maintained constant at about 3200° C. and the time constant—12 min. It would seem as if the resistance to grain growth had decreased in the region between 2.5 and 4 per cent. thoria. This apparent decrease in resistance to grain growth is readily explained by the germinative temperature laws.[17–26] The increase in resistance to grain growth is continuous with increase in the amount of thoria, according to the dotted portions of the curve shown in Plate 1. The germinative conditions have not produced exaggerated growth of grain up to about 1.5 per cent. thoria,

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* Off-setting is the transverse displacement of a section of the filament wire. The displacement occurs at places where a grain boundary occupies the whole cross-section of the wire.

nor above about 4.5 per cent. thoria, so that the normal curve representing equilibrium grain size for the given time and temperature conditions would be independent of the exaggerated grain growth portion. These changes in grain size due to variation in the quantity of thoria can be seen plainly in the micrographs. Figs. 10, 11, 12, 13, and 14 are micrographs of the tungsten ingots containing respectively 1, 2, 3, 4, and 5 per cent. thoria. The ingots were all sintered at 3200° C. for 12 min. Figs. 15, 16, 17, 18 and 19 are micrographs of the same samples containing 1, 2, 3, 4, and 5 per cent. thoria respectively, at a higher magnification.

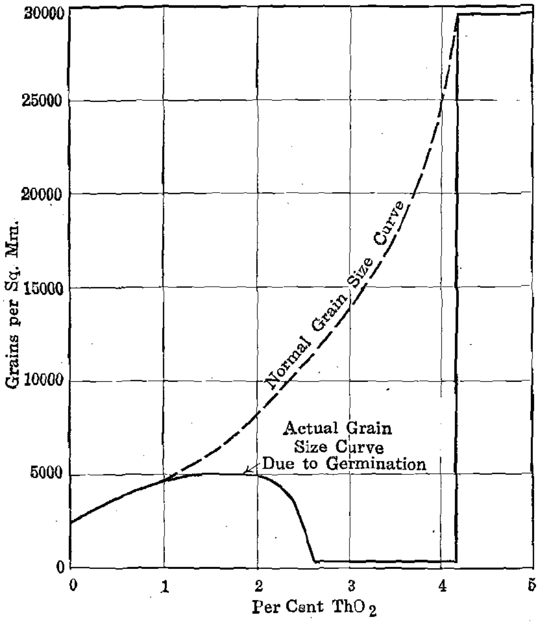

Plate 1.—Variation in grain size of tungsten ingots with variation in thoria. Heated 12 min. to mean temperature of 3200° C.

The micrographs at the higher magnification show very plainly not only the quantity of thoria, but its distribution. The thoria is present in small spherical globules in all of the samples. The number of globules is much greater in Fig. 19 than in Fig. 15. The size of the globules, however, does not vary in a marked degree.

The explanation for these coarse-grained structures with increase in resistance to grain growth is as follows: Let us take, for example, the sample containing 3 per cent. thoria. When it was heated to a mean temperature of 3200° C. by the passage of an electric current in an atmosphere of hydrogen, the axis of the comparatively long tungsten ingot would be its hottest portion. A temperature gradient would obtain from the axis to the surface of the ingot. With 1 per cent.

thoria present, even the surface of the ingot, when heated to a mean temperature of 3200° C. would permit the free growth of adjacent grains. With 3 per cent. thoria, however, the resistance to grain growth will have been sufficiently increased so that the portion of the tungsten ingot near the surface will be below the temperature of free grain growth. The portions nearer the axis, being hotter, will permit grain growth to take place. There will be a sharp boundary line between that portion of the ingot in which grain growth could take place and that portion in which growth was not possible (or at least could take place but slowly) under the existing conditions. The grains in the growth range will be larger than those in the inert range. At the boundary between the growth and inert grains, the former, because of their larger size, will be able to absorb the latter, which cannot coalesce with one another because they are (1) too nearly the same size, and (2) too cold. Adjacent grains in the growth range away from the boundary between the growth and inert grains, cannot coalesce freely with one another because they are too nearly the same size. Their size, however, is considerably greater than that of the grains in the inert region. The grains at the boundary between the inert and growth regions become germinant grains. They increase their size by absorption of the inert grains until they are much larger than the hotter grains in the growth region, which they then absorb. The resulting structure will be an extremely coarse-grained one, with a general tendency for the grains to be radial, much as if the ingot had solidified in a mold from the molten condition.

In the sample containing 2.5 per cent. thoria, germinant grains were formed first at a lower temperature, because the resistance to grain growth would be less with 2.5 per cent. than with 3 per cent. thoria. With the same average temperature, the germinant grains in the 2.5 per cent. thoria sample would first form nearer the surface of the sample. This is exactly what was observed. With the sample containing 4 per cent. thoria, the germinant grains would first form in a hotter portion of the ingot. It was observed that the germinant grains did form initially in the 4 per cent. thoria ingot practically at its axis. This indicates that with the 4 per cent. thoria ingot at a mean temperature of 3200° C. the region of free grain growth occurs only in the hottest portion of the ingot and all other parts of the ingot are in the inert range. With 5 per cent. thoria, no part of the ingot is sufficiently hot with a mean temperature of 3200° C. for free grain growth to take place in a time period of 12 min., so the whole cross-section of the ingot is in the inert range. The samples containing 1 and 2 per cent. thoria, at a mean temperature of 3200° C. were sufficiently hot for the whole cross-sections to be above their germinative temperatures, that is, to be entirely within the growth range. It will be noted that the 2 per cent. thoria sample is finer grained than the 1 per cent. sample and the sample containing 5 per cent. thoria is the finest grained of them all. It will be noted also in these samples that where grain growth has taken place the thoria globules have not apparently changed in position. For example, in Fig. 17 a portion of one grain only is shown. The thoria globules, however, are distributed within this grain in a manner very similar to the distribution of the thoria globules in Fig. 19, which consists of a fine-grained structure with the thoria globules largely situated at the boundaries of the small grains.

Some calculations indicate that the difference in temperature between the surface of the tungsten ingot ¼ in. (6.35 mm.) square and its axis is about 150° C. when a mean temperature of 3200° C. is maintained by the passage of electric current through the ingot.

By heating these samples for various time periods, the history of grain growth can be studied. In the growth range, an apparent equilibrium grain size is reached within a few minutes at a high temperature, say 3200° C. At lower temperatures around 2600° and 2700° C. a longer time is needed in order to produce an apparent equilibrium grain size. Under germinative conditions, if the germinative temperature is high, the velocity of grain growth is rapid, and if the germinative temperature is low, the velocity of grain growth is slow. At a temperature of 2600° C. the rate of grain growth was only about one-twentieth of the rate at 2300° C.

The sintering together of the individual particles of tungsten, or their welding, is an entirely distinct operation from their coalescence. At a temperature of about 2400° or 2500° C., sintering takes place and is complete within a few minutes. Most of the voids have been closed and most of the shrinkage of the ingot takes place during the sintering. With increase in temperature, these small grains formed by the sintering operation commence their growth. If no germinative temperature condition is encountered, the grains will increase in size with increase in temperature, and up to a certain point with increase in time of exposure. Germinative conditions, however, may change the order of grain size enormously.

When the germinative temperature is maintained at a point about midway between the axis and the surface of an ingot, the large germinant grains grow both toward the surface, absorbing the inert grains, and toward the axis, absorbing the grains which are in approximate equilibrium with each other. At the boundary lines between two large germinant grains and a fine-grained region, there will always be, if the fine-grained region is considered as a sea and the coarse-grained region as land, a bay at the intersection of the two large grains with the fine-grained region. This indicates that the two large grains may be competing for the small grains at this point. The forces of attack will act in different directions and a sort of neutral zone in which grain growth is less rapid than normal will obtain. Fig. 20 shows this phenomenon.

Tungsten ingots made up of an aggregate of large and small grains are not easily worked. They are apt to crack at low temperatures along the grain boundaries, between the large grains or at boundaries between large- and small-grained areas.

EFFECT OF CHANGE OF TEMPERATURE ON THE STRUCTURE OF TUNGSTEN INGOTS

Since a temperature of 3200° C. produces the germinative conditions at the axis of a tungsten ingot containing 4 per cent. thoria, a fine-grained structure would be produced by heating to temperatures lower than about 3150° C. The germinative temperature of a tungsten ingot containing 5 per cent. thoria, or more, is theoretically above the melting point of tungsten, so it is non-existent.

A tungsten ingot containing 2.5 per cent. thoria has its germinative temperature near the surface of the ingot when a mean temperature of 3200° C. is maintained. Therefore, if the temperature were lowered to about 3100° C., the germinative temperature would occur near the axis in this sample. If, however, the temperature could be increased to 3300° C. before germination had begun during heating, a fine-grained structure would result, which would correspond to the normal equilibrium grain size in the growth range for the given time and temperature conditions.

With the sample containing 1.5 per cent. thoria, a mean temperature of 3200° C. was sufficient to make every part of the cross-section of the ingot above its germinative temperature. If the temperature could have been lowered to, say, 2900° C., germinative conditions would have obtained in this sample with the resultant production of a coarse-grained structure. The germinative temperature of tungsten ingots containing 0.75 per cent. thoria is about 2550° to 2650° C. This germinative temperature is so low for tungsten that the individual particles of the tungsten powder must be very small or the formation of large grains at the germinative temperature will be defeated.

Some tungsten powder was prepared consisting of grains or particles very much smaller than the size usually employed. Experiments were made with tungsten ingots pressed from this very fine tungsten powder. It was found that the germinative temperature occurred at about 2600° C. The time necessary to produce a marked coarsening of the grain at the germinative temperature was very much longer in this material than with material whose germinative temperature was in the neighborhood of 3200° C.

Fig. 21 represents the fracture of one of these tungsten ingots heated for 20 min. at a temperature near 2600° C. Fig. 22 is the fracture of an ingot treated at a temperature of about 2600° C. for 30 min. In both of these photographs, the coarse-grained areas can be seen in the region midway between the axis and the surface of the sample. The axis itself is fine-grained and the surface is fine-grained.

Fig. 23 is a fracture of one of these tungsten ingots which was heated quickly to a temperature of 3200° C. and held for 30 min. The structure is fine-grained and uniform.

Fig. 24 is a micrograph of a tungsten ingot held at 2600° C. for 20 min.

Fig. 25 shows the cross-section of an ingot heated to about 2600° C. for 30 min. The fine-grained portions near the surface in both these samples is finer grained than that near the axis.

Fig. 26 is a longitudinal section through section *A-A* of the same ingot as Fig. 25.

Fig. 27 shows how the germinative temperature conditions produce coarse-grained structures at the ends of the ingots which are held in the electrodes. There is a temperature gradient at the ends of the ingots, because the electrodes are either directly or indirectly water cooled so that a change of temperature of at least 2000° is maintained between the portion of the ingot held in the electrode clamp and the hottest portion. Fig. 27 shows where the germinative temperature existed. To the left of the coarse-grained region may be seen the inert region, and to the right the gradual change of the coarse-grained area to a normal fine-grained structure. It is needless to say that the sintering temperature of this ingot was far above its germinative temperature.

Fig. 28 shows the structure of a tungsten ingot, heated quickly past the germinative temperature and held there for 30 min. Any intermediate structure between the coarse-grained structure formed at the germinative temperature and the normal fine-grained structure formed by heating quickly through the germinative temperature range can be produced by varying the time in which the ingot remains in the germinative temperature range during heating—that is, by varying the rate of heating.

Figs. 29 and 29*A* are micrographs of a tungsten ingot which was held for 20 min. at 2600° C., and then heated quickly to 3200° C. and held there for 10 min. The grain size of this sample is very much coarser than the normal grain size formed by rapid heating through the germinative temperature range and much finer than the grain size which was formed by a long sojourn in the germinative temperature range.

The smallest grain size that can be produced in an ingot will depend upon the size of the tungsten particles composing the tungsten powder. At the time these particles weld themselves together (sinter), the smallest grain size which it is possible to obtain from this tungsten powder will result. A tungsten ingot in this condition, however, finds very little use in the industries. It is too fine-grained to be easily amenable to the tungsten working process, especially when it is desired to secure a great amount of permanent deformation. For example, a tungsten ingot treated in this manner can be swaged and drawn only with the greatest difficulty from an ingot ¼ in. square to a small drawn wire. Such a metal is so hard that the diamond die wear may be 50 times as great during drawing as that of tungsten ingots composed of larger grains.

By heating the tungsten ingot just above the germinative temperature, the smallest grain size in the growth range is produced. It is only in the growth range that the tungsten ingots lend themselves readily to the mechanical working process. As the temperature increases, the grain size of the tungsten ingots increases up to the melting point of the tungsten.

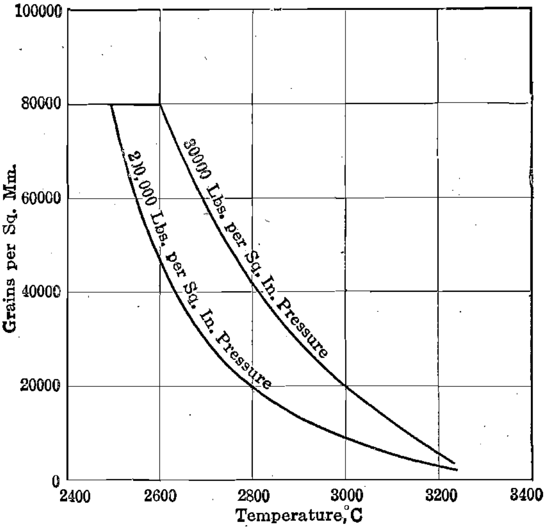

PLATE 2.—GRAIN-SIZE TEMPERATURE CURVES OF TUNGSTEN BRIQUETS. FIGURES REFER TO INITIAL PRESSURE USED IN BRIQUETTING. TIME OF HEATING, 12 MIN.

The relatively coarse tungsten powder ordinarily employed in the manufacture of tungsten ingots is sufficiently coarse grained to mask the germinative temperature phenomena in the 0.75 per cent. thoria ingots during sintering. In these ingots, the grain size starts at a minimum at about 2600° C. and gradually increases up to the melting point. A typical set of results is shown in Plate 2. The curves show the variation in grain size in tungsten ingots due to variation in the pressure to which the tungsten powder is subjected, and to variation in the temperature to which the tungsten is heated. We may call a pressure of 30,000 lb. to the square inch a normal pressure. The grain size of an ingot sintered for 12 min. at 2500° C. will be about 80,000 grains per square millimeter. At 2600° C., the grain size is the same. This indicates that below 2600° C. tungsten containing 0.75 per cent. thoria is in its inert range. The grain size will become progressively larger as the temperature is increased above 2600° C., so that at 3200° C. a grain size of 5000 per square millimeter or less will usually result with tungsten metal containing 0.75 per cent. thoria.

By increasing the pressure used in making the compressed tungsten slug to 200,000 lb. per square inch, the grain size becomes larger for any given temperature of treatment, as shown in Plate 2. The use of 200,000 lb. pressure in practice, however, is not only an extremely difficult task to perform but it is absolutely unnecessary for the accomplishment of any given purpose; for example, any result produced with 200,000 lb. per square inch pressure can be duplicated at a higher temperature with a smaller pressure.

The tungsten powder is partly amorphous and partly crystalline. The crystalline particles are probably strain-hardened by the pressure, but these strains play little part in the grain growth. They are removed before sintering takes place. The reason that high pressure tends to make larger grains is that the tungsten particles are pressed into more intimate contact, thus facilitating grain growth.

By comparing Plates 1 and 2, it will be observed that the increase in resistance to grain growth of about 4 per cent. thoria is equivalent to the decrease in resistance to grain growth due to lowering the temperature about 300° C.

CHANGES IN GRAIN SIZE DUE TO INCOMPLETE FUSION OF TUNGSTEN INGOTS

Tungsten ingots containing about 0.75 per cent. thoria remain comparatively fine grained even up to the melting point. Fig. 30 is a micrograph of one of these ingots in which the central portion has fused and resolidified and the outer portion has remained entirely in the solid state. Fig. 30*A* represents the same structure at a higher magnification. We have been taught that when fused metal at the time of solidification is in contact with crystalline metal of the same composition, this latter will act as a nucleus about which the molten metal will crystallize and assume the same orientation as the crystalline nucleus. In Fig. 30, when the tungsten was molten, it was in contact with hundreds of the small grains composing the unfused portion of the ingot. When solidification began, why did not each one of these small differently oriented grains act as a nucleus for the crystallization of the molten tungsten? They probably did. If the crystallization of the fused tungsten began from many points with different orientation, why was the resulting structure so coarse-grained as that shown in Fig. 30? The answer in my opinion is as follows: When a molten metal is in the process of solidification, it is in its most susceptible condition for grain growth. In other words, the possibility of one grain absorbing adjacent grains in an almost inconceivably short time is greater at the melting point of a metal than at any other temperature. Let us suppose that the crystallization of this fused tungsten began at the contact between the fused and the unfused portion. This must necessarily have been the case. The small grains would grow axially and if no grain growth could take place during the solidification, each of these grains would form a wedge, the small end being at the axis of the ingot. This has not taken place. Some of these grains having advantages over their neighbors, have acquired crystalline material from the molten metal faster than others. These more vigorous grains have shot out arms perpendicular to their axes of growth and have absorbed not only the molten metal which should have gone to some of the other nucleii, but also some of the crystalline material which had solidified on other nucleii. In this manner only a few of the more vigorous grains have survived.

Fig. 31 shows that if the original structure of the ingot is coarse-grained instead of fine-grained, the fused central portion will resolidify strictly in accordance with the laws of the initial nucleii. For example, it will be seen that every grain in the unfused portion has its continuation in the portion which fused and subsequently resolidified. There is no reason to suppose that at the beginning of solidification of Fig. 30, crystallization did not start from each and every small grain which was in contact with the fused metal. This leads us to the conclusion that, in cast metals, the grain size does not depend entirely on the number of nucleii from which crystallization begins, but that grain growth takes place at a remarkably fast rate during solidification.

TUNGSTEN AND CARBON

Tungsten in the presence of carbonaceous gases at high temperatures, or in contact with solid carbon at high temperatures, forms tungsten carbide, generally W_2C. Tungsten containing even small amounts of tungsten carbide at the boundaries of the grains is extremely brittle and fragile both at room temperature and at high temperatures. It has not been possible to mechanically work tungsten ingots containing tungsten carbide as a net work. Fig. 32 is a micrograph of a tungsten ingot made by adding 0.9 per cent. carbon in the form of lamp black to the tungsten powder before pressing. The tungsten carbide is the lighter constituent surrounding the grains of pure tungsten. This material is brittle, both hot and cold.

When too much carbon is added, the tungsten ingot, especially if thoria is present in the tungsten powder, will burst during the sintering operation. Fig. 33 is a photograph showing one of the bursted ingots containing carbon and a normal tungsten ingot without carbon.

If a piece of tungsten metal is heated to a temperature of 1800° C., in contact with solid carbon or in the presence of carbonaceous gases, tungsten carbide forms readily at the surface of the sample. Carbon containing gases will act more quickly and at lower temperatures than solid carbon. Hydrocarbons may form tungsten carbide in contact with metallic tungsten at temperatures as low as 1100° C. Fig. 34 shows a piece of tungsten that was heated for 1 hr. in an electric furnace in an atmosphere of CO. The lighter portion is the tungsten carbide which forms on the surface. The darker portion is pure tungsten. Fig. 35 shows a piece of tungsten metal heated for 1¼ hr. at a temperature near 1800° C., the tungsten being placed on a platform of carbon while an atmosphere of hydrogen was maintained in the furnace. The lighter portion is tungsten carbide. The depth of carbonization increases with increase in time. In 1¼ hr. the depth of carbonization was 0.21 mm. and in 3 hr. 0.30 mm. at 1800° C.

It has been found possible to work tungsten mechanically containing a considerable amount of tungsten carbide when the carbide was made separately, powdered, and added to the tungsten powder before pressing. Tungsten ingots containing tungsten carbide made in this manner may be mechanically worked when the carbon content is far in excess of that necessary to make the tungsten brittle ordinarily. When added in this way, the tungsten carbide does not surround the tungsten grains.

General Considerations Regarding the Working Properties of Tungsten

The sintered tungsten ingots are usually comparatively fine grained, say from 1500 to 5000 grains per square millimeter. It is considered desirable at present to keep the grain size of the ingots comparatively small because the inherent resistance to grain growth will usually give better life properties to the tungsten wire when used for lamp filaments. It is easily possible, however, to get the grains too fine for proper mechanical working. The more thoria or other non-metallic materials present, the more does fineness of grain militate against the mechanical working. With 0.75 per cent. thoria, which is the amount ordinarily used, if the grain size in the ingot is more than about 7000 per square millimeter, the metal will be extremely hard to work, especially in the smaller sizes. It is usually advisable to keep the grain size below 6000 per square millimeter. Tungsten ingots have been worked containing as high as 30,000 grains per square millimeter, but the metal is so hard that it is impractical to work it as a commercial process.

With very coarse-grained structures—for example, with ingots containing grains each of which measures several square millimeters in area—the first stages of the mechanical working process are very apt to break the metal. If one of these coarse-grained ingots passes the first eight or ten pairs of swaging dies, the rest of the working process is comparatively easy—in fact, easier than the working of the tungsten rods made from finer-grained ingots.

If it is desired to produce ductility in tungsten with a comparatively small amount of working, the following processes may be resorted to:

1. The working should be carried on at as low a temperature as possible.
2. The grain size of the ingot should be as small as will permit mechanical working without causing excessive trouble.

Either method, or both of the methods carried out simultaneously, will produce the desired effect, but the resulting product, which has been made ductile with a minimum amount of mechanical working, is not a suitable material for further mechanical working. It will be so hard that it will wear the dies unduly, and, furthermore, it will break and split easily during the working. What is more apt to be desired than the production of ductility in tungsten with a comparatively small amount of mechanical working is the possibility of working the tungsten a great deal before it becomes too hard and intractable for further working. This end is accomplished in several ways:

1. The grain size of the sintered tungsten ingot should be less than about 6000 grains per square millimeter.
2. The working temperature at the beginning of swaging should be very high, say 1700° C.
3. The initial swaging temperature may be considerably lower and the swaged tungsten rod can be subjected at intervals to a heating process which will cause the distorted grains formed by working to change into equiaxed grains, thereby forming a structure usually somewhat coarser-grained than that of the ingot. The equiaxed tungsten rod is then capable of a greater amount of mechanical working at the proper temperature than it would have been had it not been equiaxed.

In order to produce ductile tungsten by this process, it is necessary that it be subjected to considerable mechanical work after the last equiaxing treatment. Aside from the difference in the shapes of the grains in worked tungsten which has subsequently been equiaxed, and sintered tungsten, the two are similar, that is, the ductility at room temperature which has been gained by mechanical working below the equiaxing temperature of tungsten is lost by equiaxing; it can only be regained by further mechanical working at the temperatures indicated.

If it is desired to produce a piece of tungsten which is ductile at a particular size, this may be accomplished in the following ways:

1. By starting with a coarse-grained material of such a size that it will take a considerable amount of mechanical working to reduce it to the desired size.

2. By starting with a fine-grained material of such a size that a moderate amount of mechanical working, say an elongation of 25 times its original length, will reduce it to the desired size.

3. By starting with smaller sizes than would be used with (1) and (2) and performing the mechanical working operations at temperatures as low as possible.

The reasons for the above generalities have been largely ascertained and will be considered later.

From the time the tungsten wire reaches a diameter of 0.030 in. (0.76 mm.) or less, it is handled on reels. It would not be at all convenient, at any stage in the fine-wire manufacture, to heat it to such a temperature that the fibrous structures to which tungsten owes its ductility are destroyed. Such an operation would make the wire brittle and it could not be handled on reels in lengths of a mile or more at room temperature (as is done now) but would have to be handled in comparatively short lengths. This would slow up production to such an extent that it could not be tolerated.

Let us suppose that we have two tungsten ingots made from the same tungsten powder but that one of them has been sintered at the germinative temperature and, therefore, is coarse grained. Let us suppose that the other ingot has been sintered at a temperature above the germinative temperature and consequently is fine grained. Ingots of this nature have been made, in which the grain size was 50,000 times as great in the coarse- as in the fine-grained sample. These two ingots can be worked and treated in such a manner that when small wires are produced from them, they will be practically identical in structure. Both ingots are first swaged until their length has been about doubled or has reached some other suitable length; then these swaged rods are heated in an atmosphere of hydrogen by the passage of an electric current to a temperature above that of equiaxing. The result in an individual case was as follows:

Before treatment, the grain size ratio in the two ingots was 50,000 to 1; after swaging to 100 per cent. elongation and reheating to above the equiaxing temperature, the grain size ratio was 4:1. Fig. 36 shows a longitudinal section of the fine-grained ingot after swaging and reheating, and Fig. 37 a longitudinal section of the coarse-grained ingot after swaging and reheating. After an additional swaging and reheating, the grain size of the two rods will be practically identical. This process can be used to equalize the structures of the tungsten rods when desirable. Fig. 38 is a typical example of a longitudinal section of a swaged tungsten rod. Fig. 39 is a micrograph of a similar tungsten rod after heating for 3 min. to a temperature of 3100° C.

Tungsten rods which have been worked and recrystallized are stronger than sintered rods. In the sintered rods, the grains are exactly equiaxed, but in the recrystallized rods the grains are longer in a longitudinal than in a transverse direction.

Equiaxing Temperature of Worked Tungsten

The equiaxing temperatures of worked tungsten have been determined with an exposure of 5 min. The equiaxing temperature of a swaged tungsten rod which has been reduced in area by working about 24 per cent. is 2200° C. The equiaxing temperatures are gradually lowered both as the degree of deformation increases and as the temperatures of working decrease. Both of these factors serve to give tungsten a long equiaxing temperature range. When the swaging process has been carried on so as to reduce the area by working about 90 per cent., the equiaxing temperature is 1800° C. for a period of 5 min. With the very small tungsten wires which have been reduced in area 99.99 per cent. or more, and which have been finished at a comparatively low temperature, say a dull red heat, the equiaxing temperature in a period of 5 min. will be in the neighborhood of 1350° C. The equiaxing temperature also decreases as the time of exposure increases.

Germinative Temperature Phenomena in Worked Tungsten

When most metals have their grains permanently deformed, a slight amount, say corresponding to a reduction of area of 1 per cent. or more, but usually not more than 20 or 30 per cent., germinative temperature phenomena are apt to render them coarse grained during a normal annealing process. With ordinary muffle heating, there is apt to be no temperature gradient, or at most a very slight one, and a moderate deformation will cause within the metal a strain gradient which will cause germinative grains to form at suitable temperatures with the consequent production of coarse-grained structures.

When tungsten is deformed a moderate amount, say a few per cent. reduction in area, it should form these coarse grains at its germinative temperature, and it probably would if it were heated in a muffle to a temperature of about 2300° or 2400° C. When such a piece of tungsten, however, is heated by the passage of an electric current, the strain gradient has its maximum strain near the surface of the sample (at the point of application of the deformational forces) and the point of minimum strain is at the axis of the tungsten rod. When this rod is heated by passing an electric current through it, its axis is heated to a higher temperature than its surface. The temperature gradient, therefore, serves to counterbalance the strain gradient in such a manner as to defeat the formation of coarse grains.

As the degree of deformation increases, however, the strain gradient from surface to axis of the tungsten rod becomes practically nil. The temperature gradient during heating or during a sojourn at a certain high temperature will still obtain. Germinative temperature phenomena are then apparent in worked tungsten. When a tungsten rod has been swaged from 0.250 in. (6.35 mm.) to 0.080 in. (2.03 mm.), coarse grains can be produced by maintaining the 0.080-in. rod at a temperature just above that of equiaxing for an extended period of time. Coarse grains may also be produced in these 0.080-in. swaged rods by heating gradually with electric current to a very high temperature. Fig. 40 is a micrograph of a sample of 0.080-in. swaged tungsten rod heated to a temperature of 3100° C., the time of heating being about 10 sec. Fig. 41 shows a piece of the same 0.080-in. swaged tungsten rod which was heated as quickly as possible to 3100° C. It will be noted that the sample that was heated quickly is comparatively fine grained and the sample that was heated more slowly is extremely coarse grained. The sample shown in Fig. 41 can be maintained at a temperature of 3100° C. for hours without changing its grain size noticeably. This shows that a variation in the change of rate of heating of 10 sec. may have more influence on the resulting grain size than an exposure of many hours, or even many days, at the highest temperature reached. All rates of heating requiring more than 10 sec. to reach 3100° C. will produce coarse-grained structures in these 0.080-in. rods. The heating of an 0.080-in. swaged tungsten rod to a temperature of 2800° C. in a period of 10 sec. or more will result in a coarse-grained structure.

It should be noted in this respect that the presence of thoria in swaged tungsten operates against the formation of these large grains. It should also be noted that all of the heating is done by passing electric current through the metal itself in an atmosphere of hydrogen, thus causing a temperature gradient to exist in the heated sample. These examples of rapid grain growth are with tungsten ingots containing no thoria.

At a temperature of 2750° C., the rate of heating must be 11 sec. or more in order to produce the coarse-grained structure. At a temperature of 2700° C., if the time of reaching that temperature is less than 7½ min., the structure will be fine grained, whereas if the time is more than 7½ min., the structure will be coarse grained. At a temperature of 2600° C., the time of heating to that temperature must be more than 20 min. in order to produce a coarse-grained structure. In these remarks, a coarse-grained structure indicates the very coarse-grained structure produced under exaggerated grain-growth conditions. These time periods will also change with the particular material used.

If the 0.080-in. tungsten wire is held at a mean temperature slightly above the equiaxing temperature for an extended time, large grains will form according to the regular germinative temperature law. Fig. 42 will illustrate this grain growth at constant mean temperature, and a constant temperature gradient. This is a micrograph of an 0.080-in. swaged tungsten rod which has been reheated to a comparatively high temperature. The micrograph shows that portion which was held in the electrode clip and also the portion near the clip. It will be seen that on the left of the micrograph the grains are deformed. This was obviously the coldest portion. Temperature gradients in two directions would obtain near the electrode clip. There would be an axial temperature gradient and a radial one. The portion at the extreme right of the micrograph was above the germinative temperature and this portion exhibits a rather uniform grain size throughout. At all portions, however, between the unrecrystallized portion and the extreme right of the micrograph, the germinative temperature has existed in some part of the rod. In the hotter zone, the germinative temperature has been near the surface, and in the cooler regions, near the axis of the rod. The germinative temperature areas can be seen readily in this micrograph, although sufficient time was not given for the germinative temperature regions to encroach upon the smaller grains of either the hotter or colder portions of the sample. This figure, therefore, represents the first stages of grain growth in the germinative temperature region. Fig. 43 is a micrograph of a similar sample after the germinative grains have reached a larger comparative size. Fig. 44 is a micrograph of the same sample as Fig. 43 in the hotter portion which was above the germinative temperature. It will be noted that the colder germinative temperature portion has produced larger grains than the hotter portion which was above the germinative temperature.

When an 0.080-in. swaged tungsten rod is held at its germinative temperature, it takes considerable time for the coarse grains to form on the entire cross-section of the rod. As the size of the tungsten wire becomes smaller, sometimes a few seconds at the germinative temperature is sufficient to cause the formation of coarse-grained structures. The presence of thoria also impedes the formation of large grains in these small tungsten wires.

Table 2 shows the minimum time required to change tungsten wires originally possessing fibrous structures to coarse-grained structures, and also the variation in the diameter of wire and in the temperature to which it is heated. The tungsten wires used in these experiments did not contain thoria. The thoriated wires sometimes take as long as 1000 or 1500 hr. to change into the coarse-grained structures unless the temperature be maintained at the germinative temperature. In this event, a few hours only are required to form the coarse-grained structure. It should be noted from Table 2 that tungsten wires 0.004 in. in diameter form coarse-grained structures easily, whereas tungsten wires 0.005 in. in diameter or greater seem reluctant to form the coarse grains when the temperature is maintained constant either at or above the germinative temperature. The coarse grains form more quickly at the germinative temperature than they do at higher temperatures, but there is a marked difference in time required to form the coarse-grained structures at the germinative temperature between the tungsten wires less than 0.005 in. (0.127 mm.) diameter, and more than 0.005 in. diameter.

TABLE 2

Diam. of Wire in Mils	Temperature in °C.	Minimum Time Required to Change to Coarse-grained Structure	
		Minutes	Seconds
3	3,100	3	0
3	2,900	2	45
3	2,800	2	0
3	2,700	0	45
3	2,650	0	30
3	2,600	0	7
3	2,550	0	20
3	2,500	0	20
4	3,100	more than 2	30
4	2,900	3	0
4	2,800	3	0
4	2,700	2	45
4	2,650	2	45
4	2,600	2	45
4	2,550	1	45
4	2,500	1	0
5	2,900	13	0
5	2,800	more than 30	0
5	2,700	more than 30	0
5	2,650	more than 28	0
5	2,600	more than 40	0
5	2,550	more than 60	0
5	2,500	more than 40	0
6	2,900	more than 30	0

In the smaller tungsten wires, say below 0.005 in. diameter, containing no thoria, it will be noted that coarse-grained structures are formed in comparatively short time periods when the temperature is raised above the germinative temperature very quickly and maintained above the germinative temperature. The coarse grains form more readily, however, in these wires at the germinative temperature than at higher temperatures. Furthermore, a slow heating through the germinative temperature range will produce coarse-grained structures very much more readily than a rapid heating past the germinative temperature range followed by a sojourn at the higher temperature. If the diameter of the wire is more than about 0.005 in., the easiest way to form the coarse-grained structures is by slow heating through the germinative temperature range followed by a sojourn at a higher temperature. It can be seen from Table 2 that it may take as long as an hour to form coarse-grained structures with wires 0.005 in. or greater in diameter either when held at the germinative temperature or above it. Coarse-grained structures in these samples, however, can be produced in a few seconds by heating the wire slowly through the germinative temperature range.

Fig. 45 is a typical example of the fine-grained structures produced in these experiments and Fig. 46 is a typical example of the coarse-grained structures.

The formation of coarse-grained structures during a gradual heating through the germinative temperature range needs further consideration here. The effect of heating so quickly that germination does not have time to assert itself in the germinative temperature range has already been considered. In the pressed tungsten slug, for example, a heating period of 6 min. can be used to attain the highest temperature without any exaggerated grain growth. Some of the swaged tungsten rods, however, and also the small drawn wire, must be heated through the germinative temperature range in less than 10 sec. to defeat the formation of the coarse-grained structures. The germinative temperature will be lower as the time of heating is increased. For example, if a tungsten wire is heated by electric current to a temperature just above that of equiaxing and held there for an indefinite time, coarse grains will be formed for reasons discussed above. If, however, the tungsten wire is held at a temperature several hundred degrees above the equiaxing temperature for a short time, a coarse-grained structure may be produced in a shorter time. If the tungsten rod is heated to a temperature far above that of equiaxing, fine grains will first form. They may change to large grains due to normal grain growth, but not by germination; or they may remain as small grains.

The equiaxing temperature is a function of the time, and consequently the germinative temperature which depends to a certain extent upon the equiaxing temperature will also be a function of the time. If we could heat a piece of cold-worked copper to 1000° C. and cool it again to room temperature, the whole cycle taking but a millionth of a second, we would find that the structure of the wire had not been materially changed nor would the properties at room temperature be changed. In other words, cold-worked copper which will recrystallize or equiaxe at a temperature of 250° C. if sufficient time be given, will not equiaxe or recrystallize in a millionth of a second at a temperature 750° higher. Experiments of this nature have been conducted on tungsten wire. The wire can be flashed to a temperature near its melting point and quickly cooled to room temperature without apparent change in its structure or properties. Recrystallization of deformed grains involves a certain time period. If the temperature is comparatively low, the time is long, and *vice versa.*

When a fibrous tungsten wire is heated at a rapid rate by means of electric current, but not so rapidly that the germinative temperature laws are masked, germinant grains will form at some temperature depending upon the rate of heating. These germinant grains should be able to absorb both hotter and colder smaller grains faster than these smaller grains can coalesce with one another. When this condition obtains, coarse-grained structures will result. The gradual rise in temperature favors the absorption of the smaller grains by the larger germinant grains. The grain growth becomes a race between the germinant grains and the smaller grains. This race is in its critical period at the beginning of germination. After the germinant grains have once acquired a size much larger than the mean size of the smaller grains, the latter are absorbed readily at any temperature in the grain-growth region.

Another example of germinative temperature conditions is shown in Fig. 47, a micrograph of a molybdenum hook used to support tungsten filaments in electric incandescent lamps. This hook was in the fibrous condition when it was put in the lamp. It has been heated in the lamp from the heat of the tungsten filament which it supported and with which it was in contact at one point only, namely, at the bottom of the loop portion. It received heat by conduction and radiation from the coiled tungsten filament. There would be temperature gradients in two directions in this wire, one axially and one radially. The combination of these two temperature gradients has produced the structure seen in Fig. 47. It will be noted that at the point of contact between the tungsten wire and the molybdenum hook, the latter is fine grained. The temperature at this point was above the germinative temperature. At points more remote from the point of contact between the hook and the filament, there is every gradation in structure from fine grained throughout to coarse grained throughout. At the contact between the recrystallized and unrecrystallized portion (shown at the extreme right of Fig. 47) the molybdenum hook is very brittle. It is ductile in the fully recrystallized region and also in the unrecrystallized region.

EFFECT OF TIME AND TEMPERATURE ON GRAIN SIZE IN TUNGSTEN

A series of experiments was carried out on swaged tungsten rods 0.075 in. diameter made from tungsten metal of three different varieties. Two of the sets of samples contained 0.75 per cent. thoria and the third set contained no thoria. The results of these experiments are given in Table 3.

This table shows plainly that the increase in grain size with increase in time is not regular. It is as regular, however, as might be expected with different samples. At the time these experiments were carried out, the writer was not aware of the extreme rapidity of the formation of germinant grains during heating. Some of the radical results indicated in Table 3 were found later to have been caused by the germinative temperature conditions during heating.

TABLE 3

Kind of Metal	Temperature °C.	Time of Exposure			Grains per Square Millimeter
		Hr.	Min.	Sec.	
(A) ¾ per cent. thoria	2,500	0	0	30	3,400
¾ per cent. thoria	2,500	0	1	0	2,700
¾ per cent. thoria	2,500	0	5	0	2,700
¾ per cent. thoria	2,500	0	25	0	2,200
¾ per cent. thoria	2,500	2	0	0	3,000
¾ per cent. thoria	3,000	0	0	30	2,300
¾ per cent. thoria	3,000	0	1	0	2,200
¾ per cent. thoria	3,000	0	5	0	2,500
¾ per cent. thoria	3,000	0	25	0	1,800
¾ per cent. thoria	3,000	2	0	0	2,300
¾ per cent. thoria	3,000	9	45	0	2,200
(C) No thoria	2,500	0	0	30	1,700
No thoria	2,500	0	1	0	1,900
No thoria	2,500	0	5	0	2,800
No thoria	2,500	0	25	0	1,000
No thoria	2,500	2	0	0	1,600
No thoria	3,000	0	1	0	900
No thoria	3,000	0	5	0	1,500
No thoria	3,000	0	25	0	1,100
No thoria	3,000	2	0	0	900
(D) ¾ per cent. thoria	2,500	0	0	30	2,000
¾ per cent. thoria	2,500	0	1	0	2,300
¾ per cent. thoria	2,500	0	5	0	1,700
¾ per cent. thoria	2,500	0	25	0	2,100
¾ per cent. thoria	2,500	2	0	0	1,800
¾ per cent. thoria	2,500	9	45	0	1,600
¾ per cent. thoria	3,000	0	0	30	1,900
¾ per cent. thoria	3,000	0	1	0	2,000
¾ per cent. thoria	3,000	0	5	0	1,900
¾ per cent. thoria	3,000	0	25	0	2,000
¾ per cent. thoria	3,000	2	0	0	1,700

The conclusions reached from the results given in Table 3 are as follows:

1. Recrystallization is complete in less than 30 sec. at temperatures of 2500° C. or above. Aside from the germinative temperature phenomena, grain growth has established an approximate equilibrium

grain size at the end of 30 sec. Further heating for 9¾ hr. increases the grain size but slightly.

2. In the tungsten rods containing thoria, the difference in grain size between a temperature of 2500° C. and a temperature of 3000° C. with the time period constant is very slight.

3. With tungsten containing no thoria, the difference in grain size for a given time between 2500° C. and 3000° C. is more marked.

Tungsten ingots during sintering also attain an approximate equilibrium grain size for a given temperature in a few minutes. This is especially true at the higher temperatures of sintering, say 3200° C., whereas if the sintering is done at a lower temperature, a longer time is necessary for an approximate equilibrium grain size to be produced.

STRUCTURE AND PROPERTIES OF TUNGSTEN FILAMENTS AFTER USE

Tungsten incandescent electric lamps are all so designed that when the lamps are used at rated voltage the temperature of the filaments will be above the recrystallization or equiaxing temperature of tungsten. This is especially necessary when the long time of heating of the filaments is considered. Since the operating temperatures of lamp filaments are in the grain-growth region of tungsten (above the equiaxing temperature and below the melting point) and since the time of heating in this grain-growth range is long, sometimes exceeding 1500 hr., the structure of the filament will change during the life of the lamp. After a short burning, tungsten wires containing thoria will possess structures similar to that shown in Fig. 48. It will be noted that the grains are longer in the direction of the working than in a perpendicular direction. During the life of the lamp, this elongation is even more marked. Fig. 49 is a micrograph of a 40-watt tungsten filament after burnout. This filament contained thoria. It will be noted that the grains have been greatly elongated during the life of the lamp. These grains are equilibrium grains so far as deformational strains are concerned. The growth in a longitudinal direction has been more rapid than in an axial direction. The underlying causes for this type of grain formation have been studied, and the following conclusions have been reached:

The spherical thoria globules existing in the tungsten ingot before working are elongated during the working operations. They do not, however, elongate as much as the metallic grains. The drawn tungsten wire will consist of metallic fibers of tungsten elongated in the direction of working and non-metallic fibers or miniature rods of thoria parallel to the metallic tungsten fibers. When this wire is heated above its equiaxing temperature, the metallic fibers will change into very small equiaxed grains, but the thoria rods, if they change at all, only break up into rows of spheres, each row occupying the same general position as the thoria rod from which it was formed. Even the breaking up of the thoria into spheres takes considerable time. After the metallic grains of tungsten have broken up into very small equiaxed grains, these coalesce with one another, forming fewer and larger grains. Grain growth will take place in accordance with the time, temperature, and resistance to grain growth. The resistance to grain growth in an axial direction will be much greater than in a longitudinal direction. In an axial direction, the thoria rods, or rows of thoria spheres, will present much more surface to resist grain growth than in a longitudinal direction, because the sides of the thoria rods will offer resistance to radial grain growth. Only the ends of the thoria rods, however, will offer resistance to grain growth in a longitudinal direction. By calculation, it can be shown that the resistance to grain growth in an axial direction due to the presence of thoria may be more than 50 times that in a longitudinal direction.

It will be seen later that the forced formation of the elongated grains in a tungsten filament makes it very much more rugged than it would have been had the grains been exactly equiaxed. Fig. 50 is a micrograph of one of the old squirted or pressed tungsten filaments after life test. This micrograph was kindly furnished by Mr. C. D. Young, of the Pennsylvania Railroad Co. It will be noted that the grains in this wire are equiaxed. This is the structure which renders tungsten extremely brittle and fragile at ordinary temperatures, whereas the structure shown in Fig. 49 renders tungsten rugged at room temperature. The reasons for this will be discussed later.

A great many different types of structure are encountered in tungsten filaments after they have been used for various lengths of time. Fig. 49 is a typical example.

The difference in resistance to grain growth between tungsten containing thoria and tungsten containing no thoria can be seen by referring to Figs. 51 and 52. Fig. 51 contains 0.75 per cent. thoria and Fig. 52 no thoria. Both wires have been heated for 10 min. to a temperature near 2850° C. These micrographs are of the natural surfaces of the wires. The boundary material of the grains volatilizes more rapidly than the crystalline material, so that the grain boundaries can be seen without etching.

If tungsten develops very coarse grains when used as a filament, it has the property of being much stiffer at high temperatures than when composed of fine grains. The wires will not sag due to their own weight even when heated to a temperature of about 2800° C. If the filament, however, is fine grained, it may sag badly due to its own weight. There is danger in producing coarse-grained structures, because if a boundary line between two grains cuts sharply across a section of the filament on a plane approximately perpendicular to its axis, one section of the filament may be displaced in a direction perpendicular to its axis, thus causing the phenomenon know as "offsetting." We now know that at the boundaries between two tungsten grains a film of amorphous tungsten exists (this assumes the validity of the amorphous theory) and that at the temperatures at which the lamp filaments are used, this amorphous film is more or less fluid and weak mechanically. This is why the fine-grained tungsten wires are weaker at high temperatures than the coarse-grained tungsten. This is also the reason that offsetting takes place at the grain boundaries traversing the whole cross-section of the wire.

It is possible to form tungsten wires which are both coarse grained and substantially non-offsetting. Fig. 53 is a micrograph of a tungsten filament that is both coarse grained and non-offsetting. It can be noted that these grains are very long in a direction parallel to the axis of the wire, and that at no place does a grain boundary extend across the diameter of the wire in such a way that offsetting can take place.

WELDABILITY OF TUNGSTEN

Many attempts have been made to weld the particles of tungsten together by working at a very high temperature. No headway has been made in this direction. To be workable, a piece of tungsten must be substantially non-porous—that is, the individual particles must have been previously welded together at a temperature near the melting point of tungsten in an atmosphere of hydrogen or other gas which is either helpful or at least not harmful to the tungsten.

Sometimes the swaged tungsten rods split during the swaging operation. Attempts have been made to weld these split portions together by working at a high temperature. The highest temperature available was 1700–1800° C. It was not found possible to weld the tungsten in this manner. Tungsten can be welded electrically at temperatures near fusion.

NOTES ON POLISHING, MOUNTING AND ETCHING TUNGSTEN

Tungsten is not an easy metal to polish. It is so resistant to the action of abrasion of the polishing powders that levigated alumina can be substituted for tripoli to advantage just preceding the rouge.

White cast iron has been used to advantage as a mounting material for small pieces of tungsten which cannot conveniently be handled without some sort of a mounting. The piece of tungsten to be mounted is put in a mold and the molten cast iron poured around it. The white cast iron and tungsten are so nearly the same hardness that flat surfaces can be produced on the tungsten during polishing. It is sometimes very difficult to mount and polish the smallest tungsten wires. This has been accomplished in a successful manner, however, on wires less than 0.001 in. in diameter. One satisfactory method is as follows: An ordinary malleable iron $\frac{3}{8}$-in. (9.5-mm.) pipe cap is planed on the closed end outside. It is then drilled on the inside to a plane parallel to the outside plane. A round cover glass is put on the inside of the pipe cap, on which are placed several pieces of the small tungsten wire to be polished. Another cover glass is placed on top of these wires, after which the opening of the pipe cap is filled with powdered glass. It is then put in a furnace, being maintained in an upright position, and heated for about 5 min. to a temperature of 800° or 900° C. It has been found in many experiments that this temperature does not affect the structure of tungsten wire. The pipe cap with contents is then removed from the furnace and the glass which has congealed is pressed tightly into the pipe cap and allowed to cool slowly. The metal portion on the end of the pipe cap is then turned off in a lathe and the glass is exposed. This glass containing the samples of tungsten is polished in the ordinary manner until the tungsten wires are exposed, and the polishing is completed in the ordinary manner.

Boiling hydrogen peroxide is used for etching most of the tungsten products. Tungsten may also be etched electrolytically with good results, using a solution of sodium hydrate for electrolyte.

Why is Fibrous Tungsten Ductile?

This question has been the subject of much work and much thought. The fact that fibrous tungsten is ductile has been known for 10 years, but no explanation has been forthcoming. The result could not have been predicted from metallurgical knowledge. The explanation has been worked out step by step. The researches which have finally resulted in an explanation also permit the postulation of some new metallographic laws relating to all metals. In making the explanation, the amorphous theory is considered valid. The explanation, however, does not depend upon the validity of the amorphous theory or any other theory; it depends on facts which have been experimentally ascertained. These researches may, in fact, be used to strengthen the amorphous theory.

Crystalline tungsten is somewhat malleable and ductile at room temperature—that is, if a single grain of tungsten can be isolated, it can be slightly deformed cold before rupturing. The cold deformation strain hardens the tungsten grain and makes it brittle, similar to this action in common ductile metals. Crystalline tungsten can also be deformed above room temperature; it is capable of more permanent deformation without becoming brittle, the higher the temperature at which the deformation is produced.

Amorphous tungsten is very brittle at room temperature. It is capable of being deformed without rupture at high temperature. Tungsten composed of small equiaxed grains is not ductile or malleable at room temperature. It may be both malleable and ductile at elevated temperatures. A fracture at room temperature shows that the break has taken place largely at the grain boundaries. Occasionally, along the fracture line, a grain is encountered which has been broken in two, but this will be caused by a local high resistance to rupture along the grain boundaries. Fine-grained tungsten can be deformed under suitable conditions (such as are outlined previously) at high temperatures.

The reasons why fibrous tungsten is ductile when cold can be stated briefly as follows: Tungsten composed of equiaxed grains is brittle at room temperature because the brittle amorphous phase at the grain boundaries permits rupture before a load sufficient to deform the malleable crystalline phase can be applied. Tungsten possessing this structure is brittle even though the crystalline material present is more capable of permanent deformation at room temperature than the crystalline portions of fibrous tungsten. Fibrous tungsten is ductile at room temperature (assuming that the fiber has been produced under proper conditions) in spite of the fact that it contains more of the amorphous phase than equiaxed tungsten. It is ductile because the grain distortion by working arranges the grain boundaries in such a manner that the resistance to rupture along them is so great that rupture is forced to take place through the deformed grains themselves. These grains, which will usually have been deformed above room temperature, will possess the ability to be further deformed at room temperature; rupture through them cannot take place without further deforming them, so the metal assumes ductility. The subject of deformation of metallic grains at a certain temperature and the increase of ductility by lowering the temperature will be treated later.

We must accept as a fact, which I have observed many times, that rupture along the amorphous planes in tungsten is much more pronounced along grain boundaries than along the amorphous slip planes in deformed tungsten grains. It is probable that the path of rupture along the amorphous slip planes in a deformed grain would be many times more intricate, involving intermeshings of submicroscopic size, than the path of rupture along the grain boundaries themselves. Whatever may be the explanation for this, we must accept it as fact. It should be kept in mind that only when the amorphous phase of the metal becomes very brittle does the fracture seek the grain boundaries. This condition obtains in tungsten at room temperature but not in the ordinary ductile metals.

Even a fibrous tungsten wire which is ductile is only malleable when the deforming pressure is applied at all points of its circumference at the same time, like the swaging die action. When one of these wires is placed on an anvil and struck with a hammer, it splits into many threads—that is, the deformed grains separate from each other.

Probably one of the major causes for rupture along the amorphous grain boundaries in tungsten, in preference to through the grains themselves, is the difference in coefficient of expansion of the amorphous and crystalline phases. Glass, a completely amorphous substance, can be cracked by unequal heating or cooling, the cracks resulting from the different degrees of expansion or contraction of different parts of the piece. If we consider that fine-grained tungsten is made up of crystalline grains surrounded by films of amorphous tungsten, then these two phases will have different coefficients of thermal expansion. At the higher temperatures, these differences are easily adjusted because of the plasticity of the amorphous phase. At lower temperatures, however, both the amorphous and crystalline phases will be very rigid. This will force the strains of unequal expansion during heating or unequal contraction during cooling, into either the crystalline or amorphous material, or both. There are several reasons why these strains at low temperatures should be taken up largely by the amorphous phase. The amorphous phase is the only one possessing continuity. The crystalline phase consists of grains none of which actually touch each other. The continuity of the system depends on the amorphous phase. Any difference in coefficient of expansion between the amorphous and crystalline phases, no matter in what direction the difference may be, must affect therefore the amorphous phase. If internal strains are set up in this manner at the grain boundaries, smaller external loads will be required to cause rupture than would be indicated by the actual measure of cohesion of the isolated amorphous phase.

To obtain a somewhat better idea of these phenomena, let us consider the properties of tungsten wires about 0.007 in. (0.18 mm.) diameter with four types of structure.

1. *The Whole of the Wire Is Composed of a Single Grain.*—Since crystalline tungsten is somewhat malleable and ductile at room temperature, such a wire could be deformed cold. Cold deformation would strain-harden the tungsten and make it more brittle, and when continued far enough would break it because of the brittleness. The permanent deformation of the crystalline tungsten would generate amorphous tungsten at the planes of slip. The hardening and embrittling would be caused by the amorphous metal.

2. *The Tungsten Wire is Composed of Small Equiaxed Grains.*—Such a tungsten wire is brittle and fragile at room temperature. It cannot be appreciably bent (except the bending which takes place within its elastic limit) without breaking. The break will take place largely along the grain boundaries. These grain boundaries consist of thin films of tungsten in the brittle amorphous condition. The amorphous phase has in reality greater cohesion than the crystalline phase at room temperature, but it is under great internal stress due to the difference in coefficient of expansion between it and the crystalline phase.

3. *The Tungsten Wire Has a Fibrous Structure.*—To obtain a mental picture of this particular structure, suppose the ingot from which the wire was made was ¼ in. (6.35 mm.) square and contained 3800 grains per

square millimeter. It is worked at temperatures below that of equiaxing so that the grains are progressively elongated from the beginning of working. When the wire is 0.007 in. diameter, the grains will have been changed into fibers the average length of which will be about 1 in. and the average diameter about 0.00002 in. The end of a given metallic fiber will usually not be contiguous to the ends of other fibers with which it is in contact. A tungsten wire with such a structure is ductile cold. It can be drawn cold, bent cold, coiled, etc. It will contain more amorphous tungsten than the fine-grained sample which was brittle. It will be ductile cold because the metal has a tendency to break along the amorphous planes at the grain boundaries and the path of rupture along these will be so great that the break is forced to take place through the crystalline material which, though not as malleable and ductile as the crystalline material before strain hardening, will still possess the properties of malleability and ductility to a certain extent at room temperature. A break through it, therefore, must cause a certain amount of deformation before rupture can take place, thus giving rise to the property of ductility.

4. *The Wire Consists of Elongated Grains Which Have Not Been Strain-hardened.*—Such a structure results from long heating of tungsten containing thoria or other non-metallic substances. The arrangement of the grains makes a long path for rupture along the grain boundaries, so a much greater load can be applied before rupture than when the metal is composed of equiaxed grains. This makes tungsten possessing an elongated grain structure stronger or more rugged than the equiaxed structure. At times, such wires even possess slight ductility at room temperature. Electric incandescent lamp filaments frequently possess these elongated grain structures and are as a consequence very rugged.

It is thus seen that the ductility of tungsten at room temperature does not depend on the quantity of amorphous tungsten, but on its arrangement. It has been stated before that the tensile strength of a tungsten ingot is about 18,000 lb. per square inch. Tungsten wire possessing a fibrous structure at 0.007 in. diameter will have a tensile strength of about 340,000 lb. per square inch. The actual cohesion is greater in the drawn tungsten wire than in the equiaxed ingot. The cohesion measured in each case is that of a system made up of what may be considered physically as two separate materials with different properties, but in the case of the equiaxed ingot the internal weakening strains predominate and in the case of the fibrous wire they are eliminated.

As an example of the action of the crystalline and amorphous phases in equiaxed and fibrous tungsten, let us suppose that a structure is made up of grains of iron which we will consider as analogous to the crystalline tungsten, the grains being bound together with very thin films of glass which we will consider analogous to the amorphous tungsten cement surrounding the grains. In the first example, let us suppose that the grains of iron are equiaxed and unstrained—that is, similar to equiaxed tungsten. This structure will assume largely the properties of the brittle glass cement. If a grain of iron could be isolated, it would be malleable, but if the structure taken as a whole is hammered, it will fly to pieces, and if broken in tension, the breaking load will be too small to force any marked permanent deformation on the iron grains. This structure will be brittle. If, however, the structure were heated to a red heat, at which temperature both iron and glass are known to be workable, then considerable deformation could be effected by hammering or rolling. Let us suppose that such a structure could be rolled or drawn while hot until the dimensions of the deformed iron grains would be comparable to the dimensions of tungsten fiber. The glass film surrounding the grains of iron will remain intact. (In the actual working of the metal, it is very probable that the amorphous films surrounding the grains are made thicker by mechanical working.) Let us further suppose that in this amount of deformation the iron grains still possess some ductility in the cold. When cold ductility tests are made, it will be found that the glass films are no longer in a commanding position and the path of rupture will be forced largely through the deformed iron grains themselves. Such a structure with the same quantity of glass present will be ductile at room temperature. It has not been attempted to make the analogy perfect, because glass does not possess the same properties as amorphous tungsten, nor does iron possess the same properties as crystalline tungsten, and glass could not be generated within an iron grain during deformation.

That the fracture in tungsten tends to take place along the grain boundaries is shown conclusively in Fig. 55. This fracture was in a coarse-grained sample. One of the cracked boundary lines in Fig. 55 extends to the surface of the tungsten ingot, and the crack does also. In one place where the grain boundary line was rather jagged, the fracture took place through a portion of the crystalline material rather than at the grain boundary. The other cracked grain boundary abuts a fine-grained portion of the tungsten ingot at which the crack stops abruptly. This shows that resistance to rupture is less along a rather straight grain boundary than around the grain boundaries of fine-grained metal. Fig. 56 shows how the crack has jumped across the crystalline portion in preference to following the jagged grain boundary line. Fig. 57 shows the abrupt ending of a crack where the grain boundary line between two large grains intersects a fine-grained area, and Fig. 58 shows that the fracture of a fine-grained area follows in general the grain boundary line. There are two or three cracks in Fig. 42, and it can be plainly seen that these cracks follow the grain boundaries.

That rupture tends to take place along the boundaries of deformed grains in fibrous tungsten can be seen from Fig. 54. That the fracture does not take place entirely along these grain boundaries is due to the fact that the resistance to rupture through the deformed grains or fibers is less in fibrous tungsten than along the deformed grain boundaries. If the deforming load is applied to fibrous tungsten longitudinally, the wire is ductile, but if applied transversely, it is brittle.

Some General Metallographic Propositions

The discussion in the preceding caption shows why fibrous tungsten is more ductile at room temperature than equiaxed tungsten. Another significant fact has been observed with tungsten, namely, that after the limit of ductility has been reached by working at, say, a red heat, the tungsten after cooling to room temperature or other lower temperatures becomes ductile at these temperatures. Experiments have been made which show that this phenomenon is common to all ductile metals. The reasons have been ascertained. The underlying reason for the loss of ductility by working a metal at a certain temperature below its annealing temperature and the regaining of ductility by cooling to some lower temperature, is that the amorphous phase of any metal will increase in cohesion on cooling, at a faster rate than the crystalline phase. Let us refer to Plate 3. This shows the general cohesion-temperature curves of the amorphous and crystalline phases of metals. The direction of the curves will have to be determined for any given metal, but the general relationship will be found similar to those given in Plate 3. The curve traced in by a continuous line represents the change in cohesion of the amorphous phase with change in temperature. The cohesion is substantially zero at the melting point of the metal and increases as the temperature decreases, reaching a maximum at absolute zero. The dotted curve represents the change in cohesion of the crystalline phase with change in temperature. The crystalline phase disappears and changes into the amorphous phase when the metal is melted. On cooling from above the melting point, the crystalline phase forms during solidification and immediately at the melting temperature its cohesion assumes a finite value many times greater than that of the amorphous phase at the same temperature. On cooling below the melting point, however, the crystalline phase increases in cohesion at a very much slower rate than the amorphous phase. At some temperature between the melting point and absolute zero (in most metals not far from 0.35 to 0.45 of the absolute melting point) the cohesion of the crystalline phase will be the same as that of the amorphous phase. This temperature I have called the "equi-cohesive temperature."[34] It corresponds in most, if not all, metals, to the lowest equiaxing temperature of the severely cold-worked metal. Just as the equiaxing temperature of the metal is increased with decrease in the time of heating, the apparent equi-cohesive temperature increases as the time of applying the load by which the cohesion is measured is decreased. If a metal is deformed above the equi-cohesive temperature and kept at that temperature, the grains will not remain permanently deformed, but will equiaxe; the properties of the metal will be different after equiaxing. If, however, the grains are deformed below the equi-cohesive temperature, they will remain permanently distorted. Below the equi-cohesive temperature, the amorphous phase has not only greater cohesion than the crystalline phase but its cohesion increases much faster with decrease in temperature.

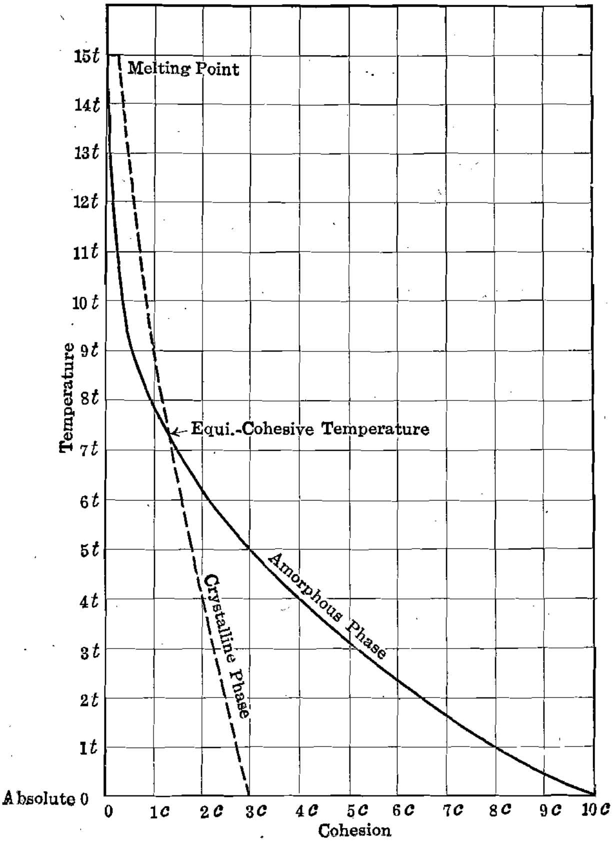

Plate 3.—Cohesion-temperature curves of amorphous and crystalline phases of any metal.

Let us suppose that Plate 3 represents the cohesion-temperature curves of tungsten and that the temperature of wire drawing is 4t°. The crystalline phase at this temperature has a cohesion of 2c and the amorphous phase 4c. The wire drawing can be continued until the load necessary to break the wire at a temperature of 4t° is not sufficient to deform the crystalline phase further. Before any deformation has taken place at $4t°$ the crystalline phase will have a cohesion of $2c$, but as the deformation continues amorphous metal will be generated at the planes of slip, and these being in a commanding position will increase the apparent cohesion of the deformed grain. Now let us suppose that room temperature corresponds to $1t°$. The cohesion of the crystalline phase at this temperature is $2.7c$ and that of the amorphous phase is $8.1c$. It will be noted that the amorphous phase at room temperature is $5.2c$ more cohesive than the crystalline phase, whereas at $4t°$ it is only $2c$ more cohesive. The limit of ductility at $4t°$ was governed to a certain extent by the difference in cohesion between the amorphous and crystalline phases. By cooling to room temperature this difference has increased $3.2c$. If now the metal is subjected to a ductility test at room temperature, a load sufficient to deform the crystalline phase further can be applied before rupture takes place; in other words, the metal is ductile and it has gained ductility because the amorphous phase increases in cohesion with decrease in temperature at a faster rate than the crystalline phase.

One point must not be confused. The limit of ductility of a metal at a certain temperature does not mean that the crystalline phase has been deformed to the greatest possible extent at that temperature. It simply indicates that the load necessary to deform the crystalline phase further cannot be applied to the metal by tension without breaking it. Even though it takes a greater load to deform the crystalline phase at a lower temperature, the increased load which can be applied by tension before rupture takes place (depending largely upon the cohesion of the amorphous phase) will be much more than the increase in the cohesion of the crystalline phase. Much more deformation can be forced on a given metal at a temperature below that of equiaxing by application of pressure than by tension. Rupturing of metals by pressure, however, must be considered as a natural modification of rupture by tension. A metal, for example, can be subjected to the highest hydrostatic pressures now available without permanently deforming it in the least. Pressure alone, therefore, will not change the external shape of a metal—that is, it will not permanently deform it. Deformation by pressure, therefore, implies a difference in pressure between two parts greater in magnitude than the cohesion of the metal to be deformed. In some cases, during the application of pressure to a metal, certain parts are actually in a state of tension and may break because of it. In other cases all parts of the metal may be subjected to pressure but certain parts will receive enough more pressure than other parts to cause permanent deformation. By utilization of the property of malleability, a metal may be deformed to a greater extent than by the utilization of ductility.

It is doubtful whether the actual limit of deformation of any crystalline metal has ever been reached experimentally. The temporary limit of deformation is always reached when the load necessary to cause it also causes the metal to rupture.

Beilby's observations on copper, gold, and silver fit in with these ideas very nicely. A description of his experiments are here given in his own words:* "Gold, silver and copper of the highest purity were the metals used. The diameters of the wires at all stages were carefully measured by a micrometer screw gage. The increases of length by wire drawing were also measured. After a final annealing, wires were drawn to as much as fourteen times their original length. Their tenacity was determined by applying a water load to the vertically hung wire. As stated above, the maximum tenacity was reached in wires drawn from *three to five times their original length.* A *hard-drawn wire shows no* general extension even under the breaking load. The broken pieces when brought together show that the small local extension which occurs at the point of rupture may amount to 1 per cent., or less, but it is purely local as proved not only by the absence of any general stretching, but also by a full series of measurements of the diameter at many points along the length. The tenacity was always calculated on the average cross-sections worked out from these measurements. After a large number of experiments had been made at the ordinary temperature, it was decided to repeat these at the temperature of liquid air or—182°. The results showed that at this low temperature the tenacity of these metals is very much increased, gold rising from 15.6 tons per square inch at 15° to 22.4 tons at −182°, silver from 25.7 tons at 15° to 34.4 tons at −182°, and copper from 28.4 tons at 15° to 36 tons at −182°. The most unexpected result was that all the hard-drawn wires stretched 11 to 12 per cent. at the lower temperature before breaking. The plasticity which had disappeared at 15° reappeared at the lower temperature. Our conclusion at the time was that the restarting of plasticity at the low temperature was due to the general increase of tenacity which enabled the wire better to resist the disruptive strains in the die."

Our discussions show that the mere increase in tenacity cannot be responsible for the ductility which Beilby observed. For example, Hadfield[38] found that the ductility of iron decreased with increased tenacity as the temperature was lowered to that of liquid air. What happened in Beilby's experiments was that the tenacity increased faster with decrease in temperature than the cohesion of the crystalline phase due to the differential cohesion between it and the amorphous phase. In Plate 3, let us suppose that room temperature corresponds to $3t°$ and liquid air temperature to $1t°$ and the curves as given represent in general the prop-

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* *Journal*, Institute of Metals (No. 2, 1911) **6**, 19.

erties of the crystalline and amorphous phases of the metals with which Beilby experimented. At room temperature the cohesion of the crystalline phase is 2.2*c*, and of the amorphous phase 5.1*c*, while at 1$t°$ the cohesions are 2.7*c* and 8.1*c* respectively. Beilby had cold-drawn the wires at room temperature till they were brittle. On cooling to −182° (1$t°$), the ratio between the cohesion of the amorphous and crystalline phases had greatly increased so that at 1$t°$ tensile load could be applied before the metals ruptured sufficiently to deform the crystalline phase further.

It will be evident, from what has been said, that the metals might be wire drawn at 1$t°$ till they became brittle at that temperature and they would then become ductile if cooled to a temperature of, say, ½$t°$.

A further experiment with a wire of aluminum copper alloy also fits in nicely with these fundamental ideas. The wire was worked cold at room temperature until it would break when bent through an angle of about 60°. A piece of wire was then immersed in liquid air and was bent double and straightened again without cracking.

Further confirming evidence is at hand and a rather complete set of experiments dealing with these general problems has now been completed and will be reported in a separate paper. The hypothesis has been sufficiently verified so that results can be predicted from it.

I have considered the application of this hypothesis to substantially pure metals and solid solutions. The application to other classes will be more complicated. Let us take steel as an example. The cohesion-temperature curves of pure iron have jogs in the crystalline curve corresponding to the allotropic points. The approximate curves for iron as well as for cementite would have to be ascertained. In annealed steel, at least three physically different substances will be present; namely, crystalline ferrite, crystalline cementite and the amorphous solution of iron and iron carbide. After cold deformation there may be five physically different constituents present; namely, crystalline ferrite, amorphous iron, crystalline cementite, amorphous cementite and the amorphous solution of iron and cementite. By knowledge of the properties of each of these constituents and their structural positions in the steel, predictions may be made regarding the physical properties of the piece of steel taken as a whole.

It should be pointed out that the metals which have the greatest possibilities for improvement by mechanical working, or those metals which should ultimately have high tensile strength whether coupled with ductility or not, should (*a*) possess high unit cohesion in both the crystalline and amorphous phases, and (*b*) the cohesion of the latter should be much greater than that of the former at room temperature. Such metals will have relatively high equi-cohesive temperatures.

In fibrous tungsten, a condition is obtained which makes the ductility greater at room temperature than that of the equiaxed tungsten. The general hypothesis of increase in ductility in metals already possessing fibrous structures by decreasing the temperatures is entirely independent of the relative ductility of the fibrous and equiaxed structures. In Beilby's experiments quoted above, for example, the ductility at −182° of the metals with fibrous structures may not have been as great as that of the equiaxed metals at the same temperature. With most, if not all, metals, however, some low temperature will be reached at which the metals in the equiaxed condition will be brittle and the same metals possessing fibrous structures which will have been produced at some higher temperature will be ductile. Iron, for example, possesses these properties at the temperature of liquid air, and tungsten and molybdenum, at ordinary or room temperature.

The amorphous phase of iron is supposed to be brittle at room temperature. It is not sufficiently brittle to cause rupture along the grain boundaries at room temperature. Liquid air temperature, however, will cause coarse-grained iron to fracture along the grain boundaries. A metal with an equiaxed grain structure cannot be very ductile at high temperature because the amorphous phase is weaker than the crystalline. Its ductility (assuming one of the ductile metals) increases on cooling to a certain point, below which it begins to become less ductile. The temperature may be lowered sufficiently to make it brittle.

Experimental proof has also been obtained showing that if a metal is worked at a certain temperature below its annealing temperature, till it becomes brittle at that temperature, it is more brittle at higher temperatures below that of annealing.

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FIG. 1.—TUNGSTEN INGOT × 152.

FIG. 2.—SWAGED TUNGSTEN ROD, 0.214 IN. DIAM. × 152.

FIG. 3.—SWAGED TUNGSTEN ROD, 0.125 IN. DIAM. × 152.

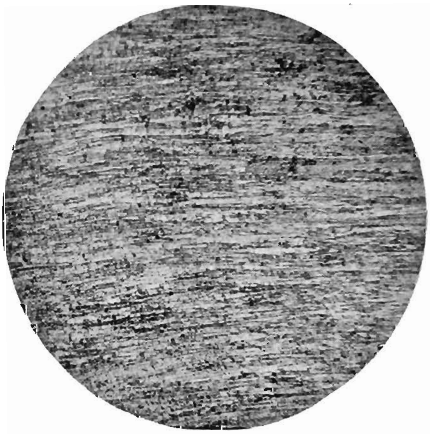

FIG. 4.—SWAGED TUNGSTEN ROD, 0.082 IN. DIAM. × 152.

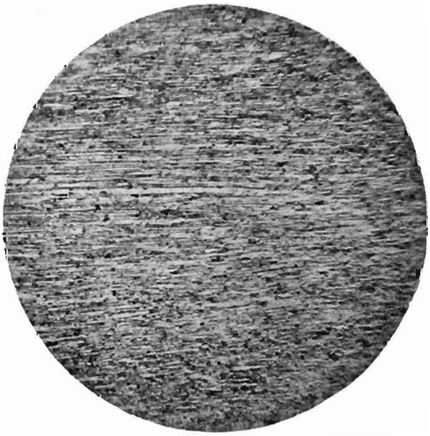

FIG. 5.—SWAGED TUNGSTEN ROD, 0.030 IN. DIAM. × 152.

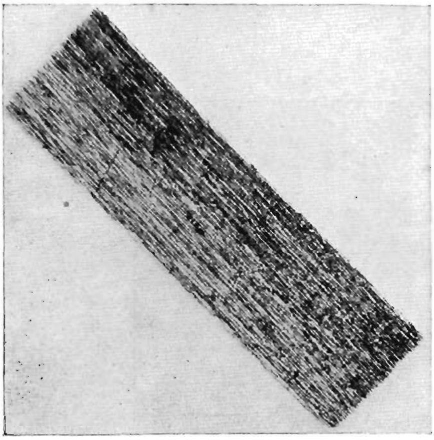

FIG. 6.—DRAWN TUNGSTEN WIRE, 0.010 IN. DIAM. × 169.

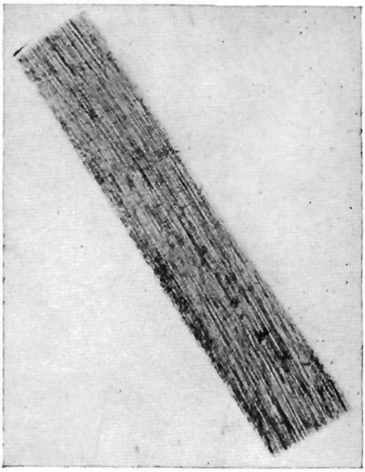

FIG. 7.—DRAWN TANTALUM WIRE, 0.007 IN. DIAM. × 125.

FIG. 8.—TANTALUM WIRE ANNEALED 5 MIN. AT 1600° C. IN VACUUM. × 125.

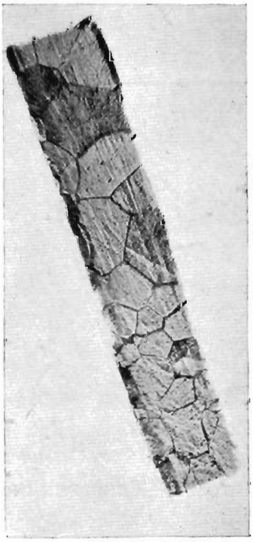

FIG. 9.—TANTALUM WIRE ANNEALED 1 MIN. IN HYDROGEN ATMOSPHERE. × 125.

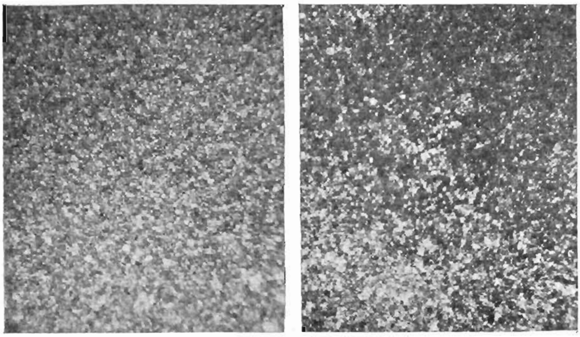

FIG. 10.—CONTAINING 1 PER CENT. ThO_2.

FIG. 11.—CONTAINING 2 PER CENT. ThO_2.

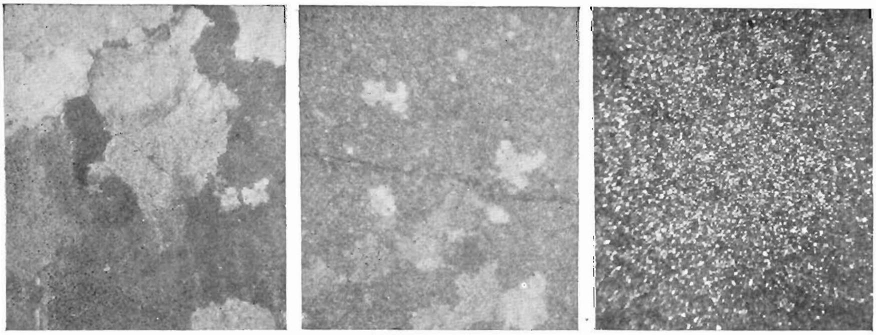

FIG. 12.—CONTAINING 3 PER CENT. ThO_2.

FIG. 13.—CONTAINING 4 PER CENT. ThO_2.

FIG. 14.—CONTAINING 5 PER CENT. ThO_2.

FIG. 10 TO 14.—TUNGSTEN INGOTS HEATED 12 MIN. AT 3200° C. × 16.

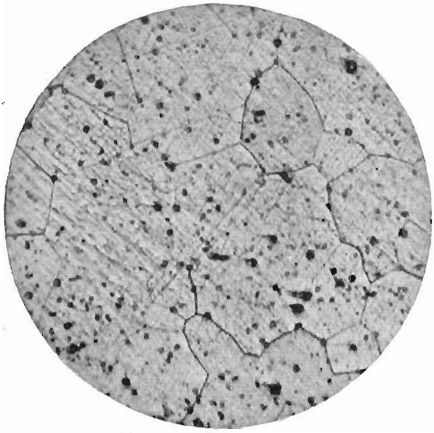

FIG. 15.—CONTAINING 1 PER CENT. ThO_2.

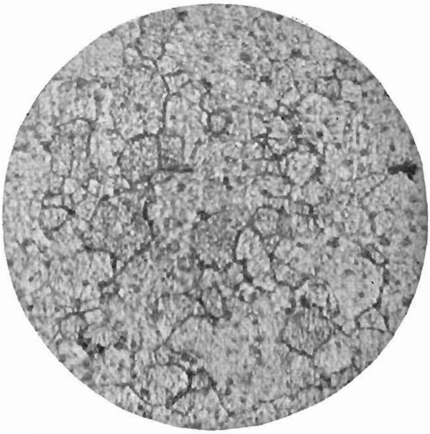

FIG. 16.—CONTAINING 2 PER CENT. ThO_2.

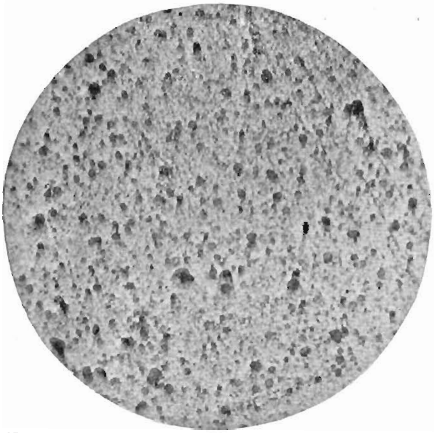

FIG. 17.—CONTAINING 3 PER CENT. ThO_2.

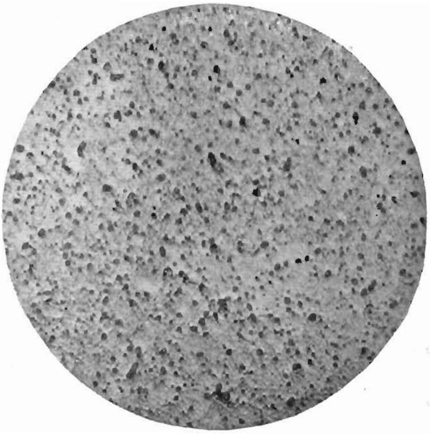

FIG. 18.—CONTAINING 4 PER CENT. ThO_2.

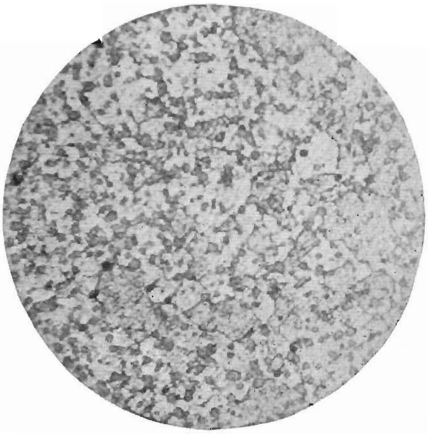

FIG. 19.—CONTAINING 5 PER CENT. ThO_2.

FIG. 15 TO 19.—TUNGSTEN INGOTS HEATED 12 MIN. AT 3200° C. × 389.

Fig. 20.—Tungsten ingot showing two large grains in position to absorb small grains. × 117.

Fig. 21.

Fig. 22.

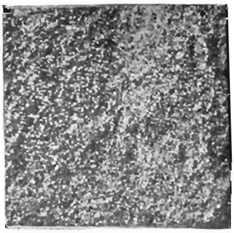

Fig. 23.

Fig. 21.—Transverse fracture of tungsten ingot containing 0.75 per cent. ThO_2, heated 20 min. at 2600° C. × 5.

Fig. 22.—Transverse fracture of tungsten ingot containing 0.75 per cent. ThO_2, heated 30 min. at 2600° C. × 5.

Fig. 23.—Transverse fracture of tungsten ingot containing 0.75 per cent. ThO_2, heated quickly to 3200° C. and held for 30 min. × 5.

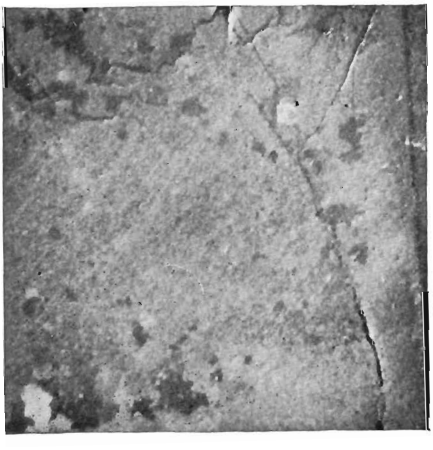

Fig. 24.—Tungsten ingot containing 0.75 per cent. ThO_2, heated 20 min. at 2600° C. Transverse section. × 9.

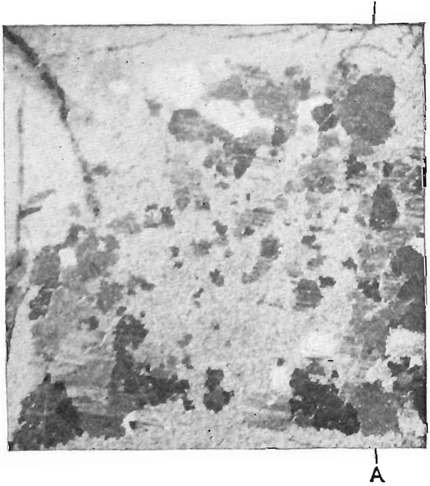

Fig. 25.—Tungsten ingot containing 0.75 per cent. ThO_2, heated 30 min. at 2600° C. Transverse section. × 9.

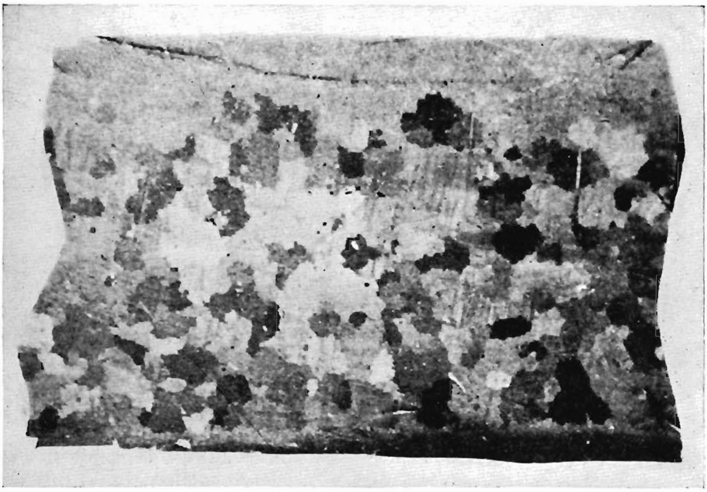

FIG. 26.—LONGITUDINAL SECTION AT SECTION A-A OF FIG. 25. × 9.

FIG. 27.

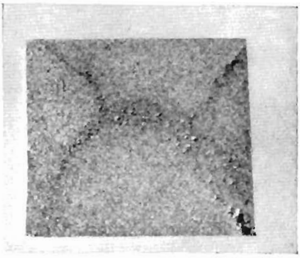

FIG. 28.

FIG. 27.—LONGITUDINAL FRACTURE OF TUNGSTEN INGOT CONTAINING 0.75 PER CENT. ThO_2. COLD END IN ELECTRODE CLAMP. × 5.

FIG. 28.—TUNGSTEN INGOT CONTAINING 0.75 PER CENT. ThO_2, HEATED QUICKLY TO 3200° C. AND HELD 30 MIN. × 5.

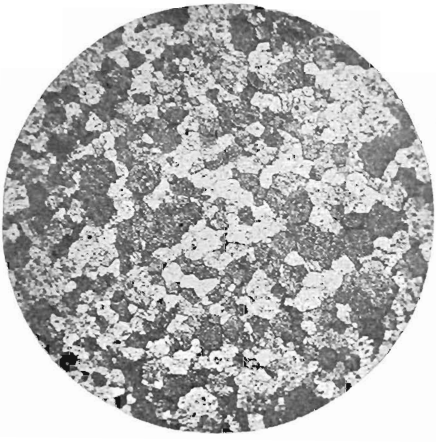

FIG. 28a.—TUNGSTEN INGOT CONTAINING 0.75 PER CENT. ThO_2, HEATED QUICKLY TO 3200° C. AND HELD 30 MIN. × 144.

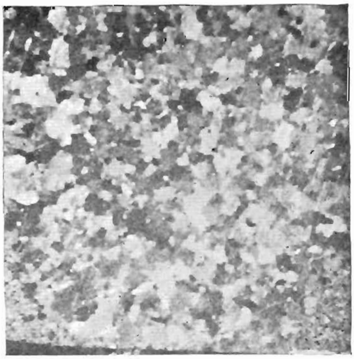

FIG. 29.—TUNGSTEN INGOT CONTAINING 0.75 PER CENT. ThO_2, HEATED 20 MIN. AT 2600° C. AND THEN HEATED 10 MIN. AT 3200° C. × 7.

FIG. 29a.—TUNGSTEN INGOT CONTAINING 0.75 PER CENT. ThO_2, HEATED 20 MIN. AT 2600° C. AND THEN HEATED 10 MIN. AT 3200° C. × 74.

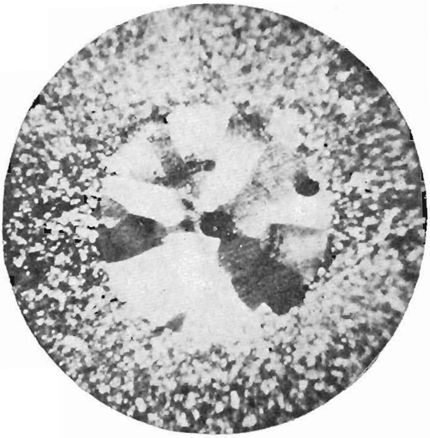

FIG. 30.—FINE-GRAINED TUNGSTEN INGOT WITH FUSED CENTER. × 15.

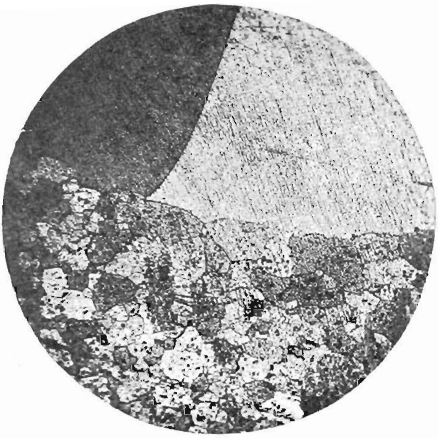

FIG. 30a.—FINE-GRAINED TUNGSTEN INGOT WITH FUSED CENTER. × 144.

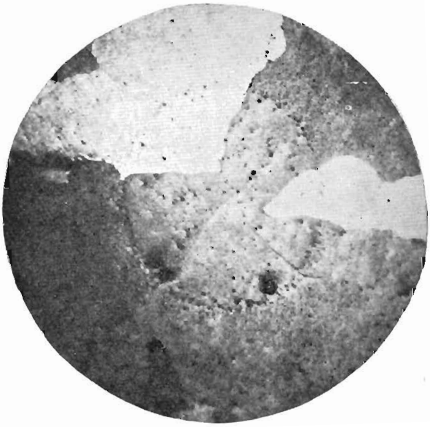

FIG. 31.—COARSE-GRAINED TUNGSTEN INGOT WITH FUSED CENTER. × 17.

FIG. 32.—INGOT WITH TUNGSTEN-CARBIDE NETWORK SURROUNDING TUNGSTEN GRAINS. × 950.

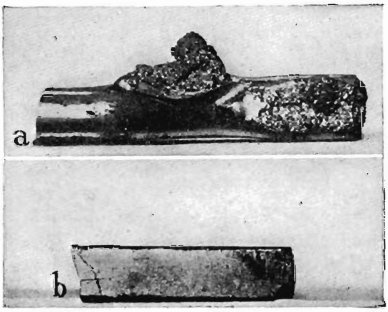

FIG. 33.—a. TUNGSTEN INGOT CONTAINING BOTH ThO_2 AND CARBON. b. TUNGSTEN INGOT CONTAINING ONLY ThO_2.

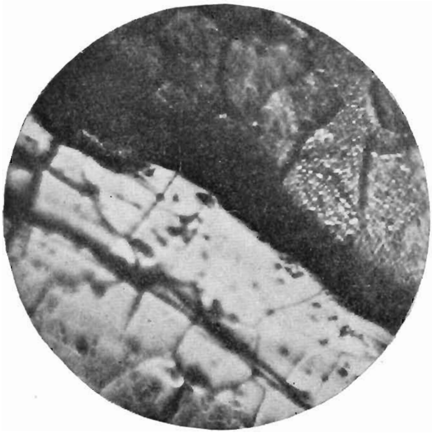

FIG. 34.—TUNGSTEN ROD CARBONIZED WITH CO. LIGHT PORTION IS TUNGSTEN CARBIDE. × 940.

FIG. 35.—TUNGSTEN ROD CARBONIZED WITH HYDROCARBONS. LIGHT PORTION IS TUNGSTEN CARBIDE. × 400.

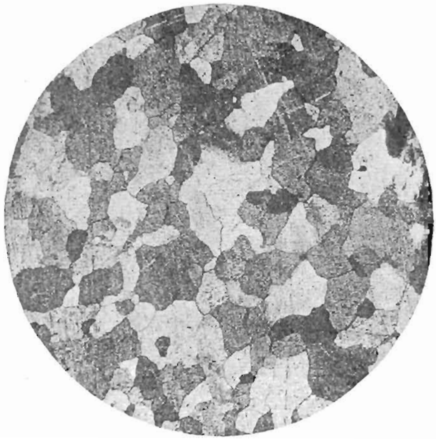

FIG. 36.—FINE-GRAINED TUNGSTEN INGOT, SWAGED AND RECRYSTALLIZED. × 74.

FIG. 37.—COARSE-GRAINED TUNGSTEN INGOT, SWAGED AND RECRYSTALLIZED. × 74.

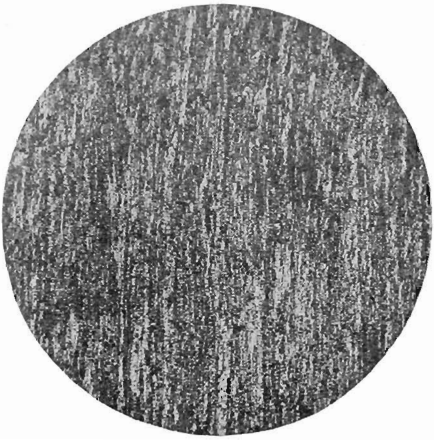

FIG. 38.—STRUCTURE OF SWAGED TUNGSTEN ROD, 0.80 IN. DIAM. × 240.

FIG. 39.—SWAGED TUNGSTEN ROD CONTAINING 0.75 PER CENT. ThO_2, 0.080 IN. DIAM. RECRYSTALLIZED BY HEATING 3 MIN. AT 3100° C. × 240.

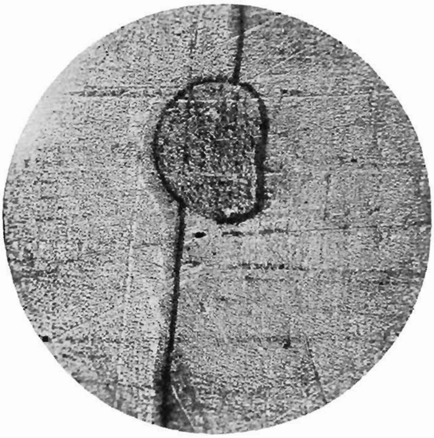

FIG. 40.—SWAGED TUNGSTEN ROD CONTAINING NO ThO_2. 0.080 IN. DIAM. HEATED BY ELECTRIC CURRENT SLOWLY TO 3100° C. × 73.

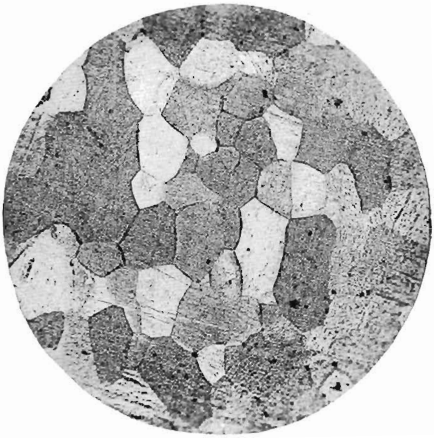

FIG. 41.—SWAGED TUNGSTEN ROD, 0.080 IN. DIAM., CONTAINING NO ThO_2. HEATED BY ELECTRIC CURRENT QUICKLY TO 3100° C. × 73.

FIG. 42.—SWAGED TUNGSTEN ROD, 0.080 IN. DIAM., CONTAINING NO ThO_2. HEATED BY ELECTRIC CURRENT 2 HR. AT 3000° C. END NEAR COLD ELECTRODE CLAMP. × 33.

FIG. 43.—SWAGED TUNGSTEN ROD, 0.080 IN. DIAM., CONTAINING 0.75 PER CENT. ThO_2. HEATED 9¾ HR. AT 3000° C. COLD END NEAR ELECTRODE CLAMP. × 155.

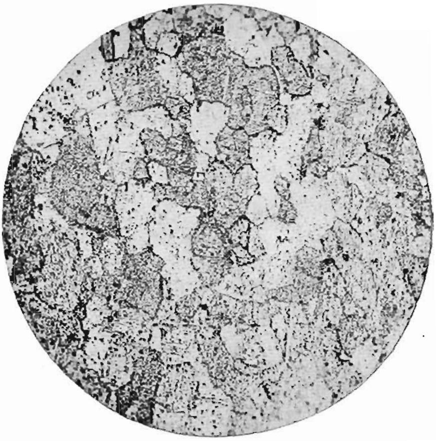

FIG. 44.—SAME ROD AS FIG. 43, IN HOTTER REGION. × 155.

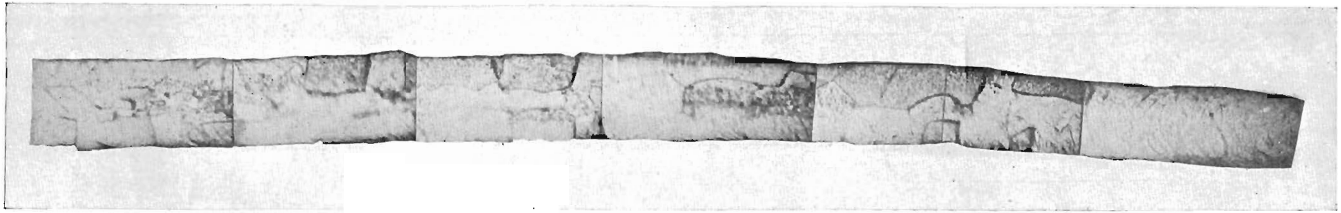

Fig. 49.—Tungsten filament 0.0015 in. diam., containing 0.75 per cent. ThO_2 after burn-out in 40-watt vacuum lamp. Longitudinal section. × 346.

Fig. 45.—Tungsten wire, 0.003 in. diam., containing no ThO_2. Heated by electric current quickly above germinative temperature. × 355.

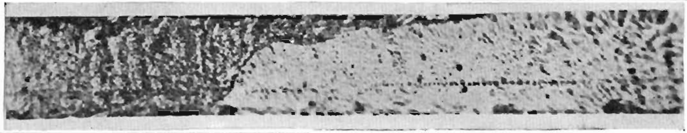

Fig. 46.—Same wire as Fig. 45. Heated by electric current slowly through germinative temperature range. × 355.

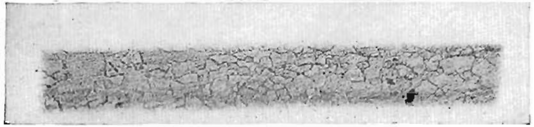

Fig. 48.—Tungsten wire, 0.007 in. diam., containing 0.75 per cent. ThO_2. Recrystallized by short heating at about 2000° C. × 130.

Fig. 47 on next page.

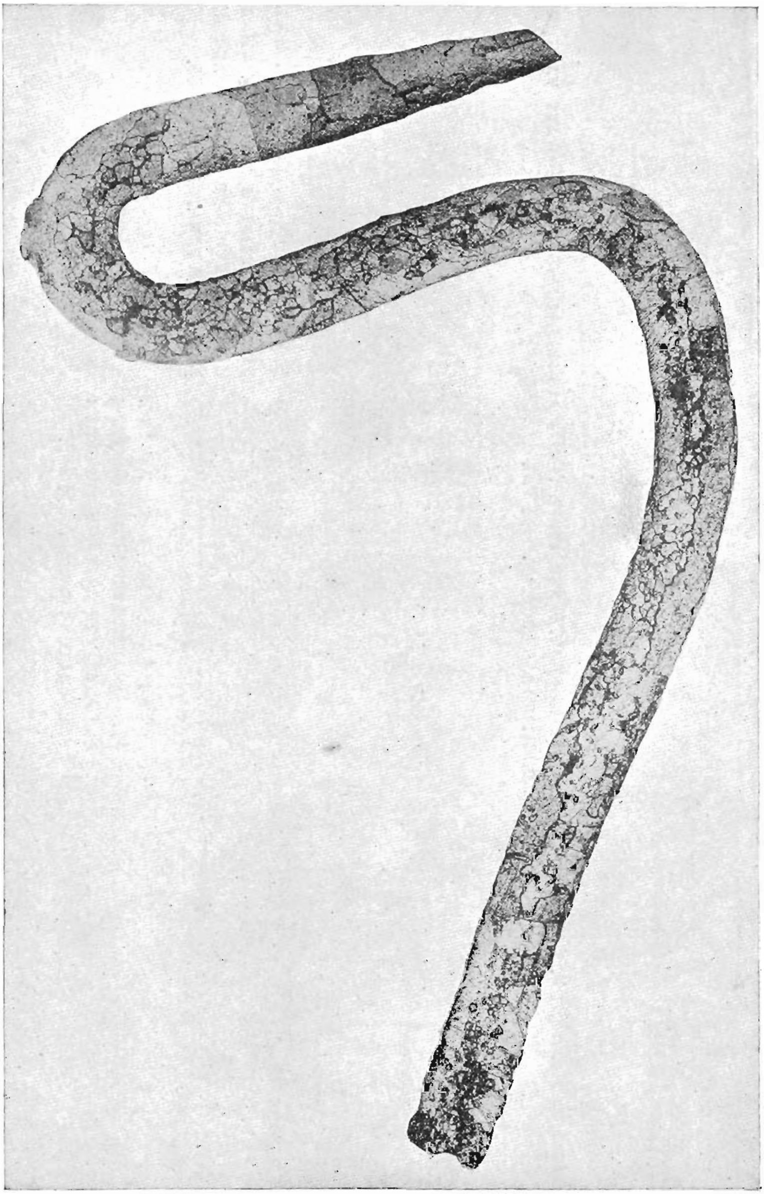

FIG. 47.—MOLYBDENUM HOOK USED AS FILAMENT SUPPORT IN 1000-WATT GAS-FILLED LAMP. LONGITUDINAL SECTION. × 104.

Fig. 50.

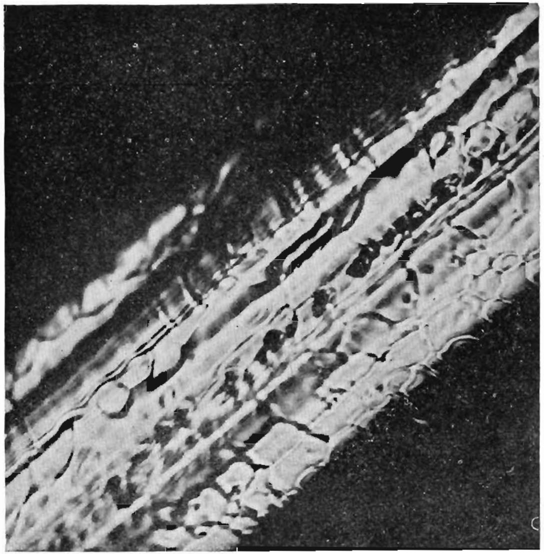

Fig. 51.

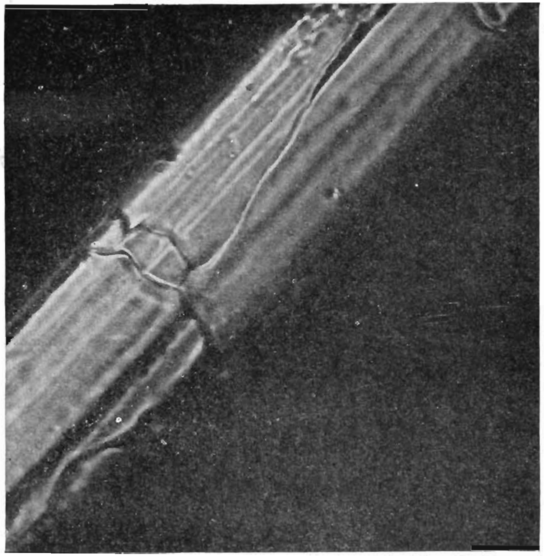

Fig. 52.

Fig. 50.—Old type "squirted" tungsten filament after burn-out in vacuum lamp. (Young.) × 325.

Fig. 51.—Tungsten wire, 0.0036 in. diam., containing 0.75 per cent. ThO_2 after 10 min. exposure at 2900° C. Natural surface. × 480.

Fig. 52.—Tungsten wire, 0.0036 in. diam., containing no ThO_2 after 10 min. exposure at 2900° C. Natural surface. × 480.

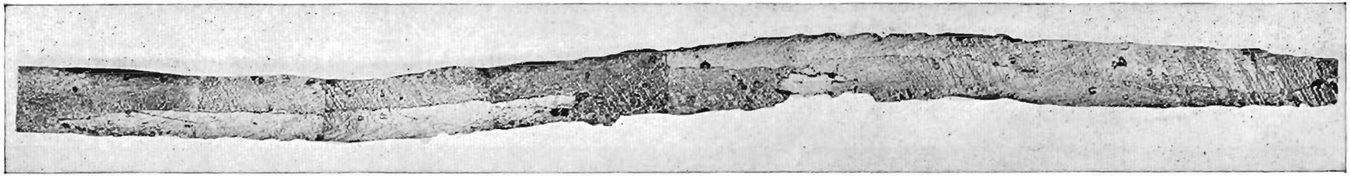

Fig. 53.—Tungsten wire, 0.007 in. diam., containing no ThO_2. Used as filament in vacuum lamp, after 66 hr. burning. Shows both large grains and absence of grain boundaries extending across whole section of wire. × 60.

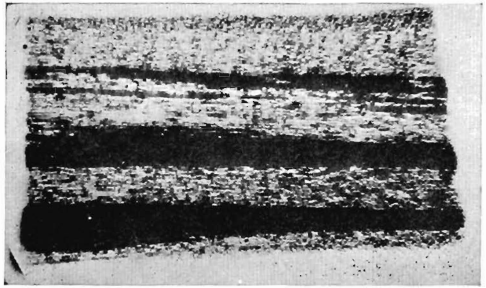

Fig. 54.—Split tungsten wire caused by "overworking" or working too cold. × 170.

FIG. 55.—CRACKS AT GRAIN BOUNDARIES IN TUNGSTEN INGOT. × 17.

FIG. 56.—CRACK PARTLY THROUGH GRAIN IN TUNGSTEN INGOT. × 50.

FIG. 57.—CRACK BETWEEN TWO GRAINS IN TUNGSTEN INGOT. STOPS AT JUNCTION BETWEEN COARSE- AND FINE-GRAINED REGIONS. × 61.

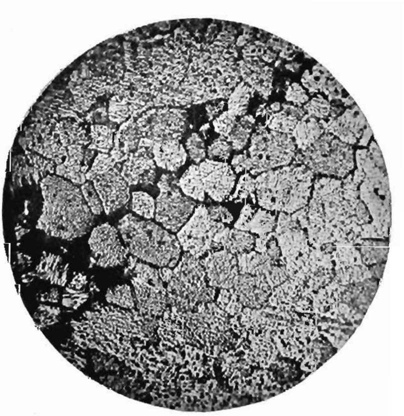

FIG. 58.—SHOWS CRACK ALONG GRAIN BOUNDARIES IN FINE-GRAINED TUNGSTEN INGOT. × 144.

DISCUSSION

Sir Robert Hadfield, London, England (written discussion*).—We have in the past known so little about tungsten that an important paper such as Mr. Zay Jeffries contributes is most welcome.

I have not much personal knowledge on the subject except as regards combinations of iron with tungsten. I put forward the results of my research on natural alloys in 1903,[1] and so far as I know the facts there presented still stand.

Singular to say, tungsten added to iron affects the electrical resistance less than any other added element. The following tables taken from research work by myself and Sir William Barrett may be of interest.

Table 1.—*Approximate Specific Electrical Resistance of Iron Alloys at Different Percentages of the Added Element*

Alloy of Iron with	Percentage of Added Element				
	1	2	3	4	6
Tungsten	15.5	16.5	17.2	18.0	18.5
Nickel	19.0	21.0	23.0	25.0	27.0
Chromium	....	....	24.0	26.5	29.0
Manganese	23.5	28.0	31.0	34.0	39.0
Silicon	....	....	46.0	53.5	69.0
Aluminium	27.0	38.0	48.0	57.0	74.0

Table 2.—*Increase in Specific Electrical Resistance (Microhms per Cubic Centimeter at 18° C.) Produced in Iron of Corresponding Purity by Alloying It with Various Percentages of the Elements Named*

Alloy of Iron with	2 Per Cent.	4 Per Cent.	6 Per Cent.
Tungsten	4.0	5.5	6.0
Nickel	6.5	10.0	12.5
Chromium	....	12.0	14.0
Manganese	15.0	21.0	26.0
Silicon	....	41.0	55.5
Aluminium	25.5	44.5	61.5

The following is the summing up of our results: Selecting the middle column of figures and dividing it by 4, we get the increase of re-

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* Received Aug. 13, 1918.

[1] *Journal*, Iron and Steel Institute (No. II, 1903) **64**, **14**.

sistance produced by the addition of 1 per cent. of the element in an alloy containing about 4 per cent. of the added element. This is shown in the next table, and side by side are shown the specific heat and the atomic weight of the elements named in the first column.

TABLE 3

Iron Alloyed with	Specific Resistance of 1 Per Cent.	Specific Heat	Atomic Weight
Tungsten	1.1	0.035	184
Cobalt	2.2	0.107	59
Nickel	2.5	0.109	59
Chromium	3.0	0.1(?)	52
Manganese	5.2	0.122	55
Silicon	10.3	0.183	28
Aluminium	11.1	0.212	27

PAUL D. MERICA,* Washington, D. C. (written discussion†).—This paper is a discussion of some of the results of a recent investigation[2] of Prof. Zay Jeffries, and of his interpretation and generalizations from these results. This work I have followed with great interest and with appreciation of its value in clarifying our views on the nature of deformation in metals and its relation to structure. It is only by such close study of the relation of structure to mechanical properties that we shall ever be able to describe the latter in terms of their ultimate elements, and thus explain what are still mysteries in the mechanical behavior of metals; experiment and thought along these lines are in my opinion of the utmost value. If I, therefore, here record some of the difficulties I have experienced in understanding some of the generalizations of Prof. Jeffries, it is only in order that perhaps from the discussion may emerge a clearer and more consistent statement of hypothesis or theory in explanation of the facts discovered by him.

Before considering the author's propositions in detail, I wish to group a few concepts and assumptions with which I here have to deal.

(1) The amorphous-metal hypothesis postulates crystalline and amorphous metal; the former, the substance of the grain in crystal, the

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* Metallurgist, U. S. Bureau of Standards.

† Received Sept. 28, 1918.

[2] Article 1: The Metallography of Tungsten, the article which precedes this discussion.

Article 2: The Amorphous Metal Hypothesis and Equi-cohesive Temperatures. *Journal*, American Institute of Metals (1917) **11**, 300.

latter existing (*a*) as a cement between grains, (*b*) in metal deformed below the recrystallizing temperature range, at the planes along which slip has occurred.

(2) The amorphous metal at the grain boundaries and that formed at a slip plane may or may not be identical in properties, but I shall make that as an initial assumption; this assumption is tacitly made by the author.

(3) Each variety of metal, crystalline (*C*) and amorphous (*A*), has its own mechanical characteristics, with three of which I shall be concerned: the ultimate tensile strength, the elastic limit, and the elongation at rupture. Neglecting the phenomenon of "necking-down" at rupture, which is a consequence of nonuniformity either of stress or of strength along the axis of a bar in tension, the stress-strain curves of *C* and of *A* metal may be assumed to have the general form shown in Fig. 1. The *C* metal has a definite elastic limit, and its properties are directional. The *A* metal probably has no definite elastic limit, but we may assume a value E_A at which the permanent deformation exceeds a certain arbitrary value; the properties of *A* are not directional, but depend undoubtedly upon the rate or duration of application of stress; *i.e.*, the *A* metal is viscous in its nature.

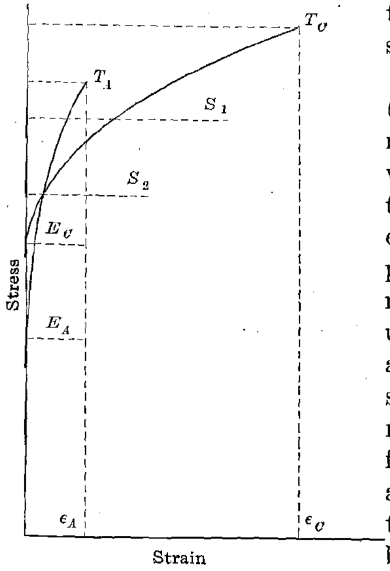

FIG. 1.—TENSILE STRESS-STRAIN CURVES FOR AMORPHOUS (*A*) AND CRYSTALLINE (*C*) METAL. ELONGATIONS PLOTTED ARE PERMANENT ONES.

(3*a*) It is likely that the elastic limit of a grain can be increased in any direction, as Tammann has shown, by the gradual exhaustion of the most accessible slip planes, but the ultimate tensile strength of the crystalline metal of a grain in any direction probably is a function only of temperature. The curves of E_C shown in Fig. 2 are to be considered as those of the undeformed metal; the values may be raised possibly up to those of T_C.

(4) The effect of temperature on the shape of these curves is to diminish T_A, T_C, E_A, and E_C, and to increase ϵ_A and ϵ_C.

(5) The tensile ductility is measured by the amount of permanent set per unit length occurring between *T* and *E*; it is undoubtedly a function of the rate of loading between these two values of stress. Exhaustion of ductility occurs, as Prof. Jeffries points out, not because the metal cannot be further plastically deformed, but because the tensile load necessary to produce the deformation will first rupture the bar. According

to Prof. Jeffries, the variations in the values of E_A and E_C, respectively, with temperature are such that for every metal (and alloy) there is a temperature (EC_E) below the melting point, such that

$$\begin{array}{rl} \text{above } EC_E & E_C > E_A \\ \text{at } EC_E & E_C = E_A \\ \text{below } EC_E & E_C < E_A \end{array}$$

This is the equi-cohesive temperature; see Fig. 2, in which E_A and E_C are shown as functions of temperature.

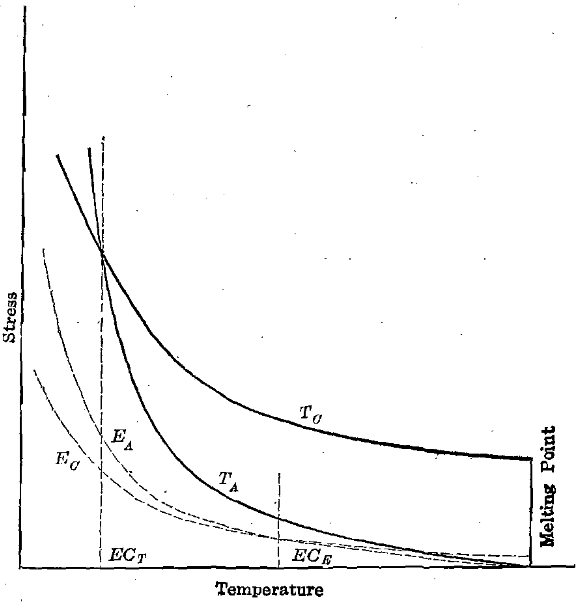

FIG. 2.—EQUI-COHESIVE TEMPERATURES AND EFFECT OF TEMPERATURE ON E (UNDEFORMED) AND T.

(6) I should like to call attention to another equi-cohesive temperature, namely, that at which the tensile strength of A and C metal are equal; such a temperature has been predicted by Rosenhain.[3] This is shown in Fig. 2; EC_E, the equi-cohesive temperature for elastic limits, and EC_T, that for tensile strength, are not necessarily equal.

(7) When a metal ruptures in tension, the path of rupture is never straight; it passes sometimes through the grains (intracrystalline) and sometimes along the grain boundaries (intercrystalline). The rupture along those portions not perpendicular to the direction of the net tension occurs, very probably, partly as a result of shearing stresses. The load necessary to cause fracture is probably proportional to the area of the path of rupture.

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[3] Walter Rosenhain: "An Introduction to the Study of Physical Metallurgy." New York, 1914. D. Van Nostrand Co.

(8) In any case, I wish to make two assumptions regarding rupture:

(*a*) That the total force to cause rupture is proportional directly to area of its path, counting sheared areas as equal in effect to those separated in direct tension.

(*b*) That the path of rupture across a grain, which usually appears to be straight, but which may involve serrations such as to increase its apparent length, is actually no longer than the path around the boundary of a spherical grain of which the apparent path is a diametral plane.

(9) In case the grain is elongated perpendicular to the general direction of stress and path of rupture, there are three potential paths of rupture:

(*a*) Across grain, through *C* metal.
(*b*) Across grain, through *A* metal (at a slip plane).
(*c*) Around grain, through *A* metal.

Rupture will occur along that path for which the product of area and unit strength is least.

I wish now to discuss on this basis some of the propositions suggested by Prof. Jeffries.

(*A*) On page 618 (Article 1), the author explains the absence of ductility at ordinary temperatures in tungsten consisting of equi-axed grains as existing "because the brittle amorphous phase at the grain boundaries permits rupture before a load sufficient to deform the malleable crystalline phase can be applied;" *i.e.*, because $T_A < E_C$ at ordinary temperatures. In his other paper (Article 2) he has found that EC_E for equi-axed tungsten was approximately 1350° C. Therefore $E_A > E_C$ below 1350° C. But, since $T_A > E_A$, therefore $T_A > E_C$ below 1350° C., a conclusion which is the exact opposite of the one assumption used above.

This contradiction may be explained, possibly, by the fact that the equi-cohesive temperatures which Prof. Jeffries determined[4] are not the equi-cohesive temperatures as he defines them on p. 311 of the same article. Reference to Fig. 1 will illustrate this; the two curves are the stress-strain curves of *A* and of *C* metal at a temperature (t), above the equi-cohesive temperature EC_E, as defined on p. 311 by the author; *i.e.*, such that $E_C < E_A$ and $t < EC_E$. Yet at a stress, S_1, the permanent set of the crystalline metal is, however, greater than that of the amorphous metal; it will be observed that it is the temperature at which, for a given stress, the permanent deformations of *A* and *C* metal are equal, which the author determines and calls equi-cohesive temperature (p. 312).

To explain lack of ductility of equi-axed tungsten along the general line of the author's explanation, we might then assume that at ordinary temperatures we are above EC_E, not below it.

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[4] *Journal*, American Institute of Metals (1917) **11**, 312.

(B) On p. 622 (Article 1), the author says, "The underlying reason for the loss of ductility by working a metal at a certain temperature below its annealing temperature" (EC probably) "and the regaining of ductility by cooling to some lower temperature, is that the amorphous phase of any metal will increase in cohesion on cooling, at a faster rate than the crystalline phase." I believe that this is not quite accurately stated; the necessary conditions to produce this effect are not so simple. They appear to me to be as follows:

(1) Since the strength of the A metal is to determine the ductility, fracture must take place through it; *i.e.*,

$$\phi_A T_A < \phi_C T_C$$

where ϕ_A and ϕ_C are the areas of the potential paths of rupture through A and C metal, respectively.

(2) But the fact is established that fracture takes place across a deformed grain; *i.e.*, through A metal, at slip planes either in tungsten (see p. 618) or in other metals below EC.

(3) Therefore $\phi_A = \phi_C$ (approx.) and $T_A < T_C$.

(4) The condition for the exhaustion of ductility is not that the "cohesion" of A and of C become equal, but that I_A is not equal to nor greater than E_C.

(5) In order that the ductility of deformed metal may reappear upon lowering the temperature, the quantity $(T_A - E_C)$ must increase with this lowering.

These conditions are represented in Fig. 2, and are quite possible ones; in fact the author's hypothesis could quite well fix the case of tungsten. But it is to be observed that condition (3) would require that in any metal within the temperature range within which ductility after deformation was restored upon lowering the temperature, fracture should occur within the same temperature range in an equi-axed grain at the grain boundaries: *i.e.*, this range is above EC_T.

But this is not true of gold, silver and copper, in which this identical phenomenon occurs. In these metals, at those temperatures, $T_A > T_C$, and the amorphous metal does not determine strength nor ductility.

(C) There is, however, a further contradiction in the author's views to which I wish to call attention. On p. 618 (Article 1) he says that fibrous tungsten "is ductile because the grain distortion by working arranges the grain boundaries in such a manner that the resistance to rupture along them is so great that rupture is forced to take place through the deformed grains themselves." In other words, equi-axed tungsten is brittle because $\phi_A T_A < \phi_C E_C$, where $\phi_A = \phi_C$ approx. When the grains are elongated, ϕ_A becomes large enough that $\phi_A T_A > \phi_C E_C$, and the fracture takes place across the grain. Now, on the basis of assumption (2) above, this fracture cannot be through A metal; for we should have, in that case, $\phi_{A_1}T_A$ (of fibrous grains) $> \phi_{A_2}T_A$ (of equi-axed grain). But $\phi_{A_1} = \phi_{A_2}$ (approx.) if the path of rupture is across grain; therefore $T_A > T_A$, an obvious absurdity. Therefore, axial fracture in fibrous tungsten must be through the crystalline metal; *i.e.*, $T_A > T_C$. But this contradicts the assumption (1) under (B), necessary to explain the regaining of ductility of deformed tungsten upon lowering the temperature.

These are some of the difficulties I have experienced in reconciling the various views of Prof. Jeffries, which lead me to wonder whether we must not examine more closely into the tacit assumptions which are made in developing such hypotheses, and also into the exact nature of deformation, before we may attempt to generalize to any too great extent. Such questions as the following must receive more thorough attention:

(1) Is amorphous metal the same in properties whether at slip planes or at grain boundaries?

(2) What is the actual shape and area of a fracture across a grain; in other words how serrated is this path?

(3) Does fracture across a grain occur within crystalline or within amorphous metal?

(4) What is the nature of the resistance to plastic deformation within a grain? This resistance would seem to be in reality a very complex function.

I believe the recent experimental results of Prof. Jeffries, as well as those of other investigators whom he has quoted, can contribute much to the elucidation of these specific questions.

J. C. W. Humfrey,* Sheffield, England (written discussion†).—Prof. Jeffries' paper gives a very complete description of the metallurgy of tungsten, the details of which afford a striking illustration of the application of scientific principles in overcoming practical difficulties of manufacture. In addition to this practical side, it deals with two phenomena of great theoretical importance and of general metallographic interest, viz.:

(*a*) Grain growth in metals.

(*b*) The significance of intercrystalline cohesion.

It is chiefly with the second of these two subjects that I propose to deal in the following remarks.

On page 617 the author states that he accepts the amorphous boundary theory, viz., that the crystals in a mass of metal are joined to one another by a cement of the same metal, in the amorphous or non-crystalline state. Agreeing, as I also do, with the validity of this theory, I am unable to accept certain of the interpretations he puts upon it, or to follow his arguments as to the manner in which his experimental data may be taken as a confirmation of its truth.

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* Admiralty Inspection Officer.

† Received Sept. 28, 1918.

The author appears to consider the amorphous cement as occupying a volume in the mass comparable with that of the crystals, and deals with the mechanical properties of the mass as if the two phases were distinct from one another and possessed little or no mutual adhesion. His comparison of the structure of tungsten with a mass of iron crystals embedded in glass is not, I think, a very happy one, nor is it indicated either by the mechanical properties of metals in general nor of this one in particular.

In a paper (which he quotes in the bibliography, No. 33) I put forward a theory based on the view that the amorphous cement owes its origin to the impossibility of two differently oriented crystals fitting exactly into one another, and the consequent existence of a certain range of atoms between them which, since they are being constrained by each crystal in a different manner, are forced to take up some irregular grouping between the two. According to this theory, there is no sharp line of demarcation between crystalline and amorphous, and the one must merge gradually into the other. The whole thickness of the disturbed layer is of molecular rather than of microscopic dimensions, and, while the amorphous envelopes are incapable of plastic deformation, yet they are capable, in consequence of their extreme thinness, of considerable distortion relative to the crystals they surround by means of elastic bending.

In all metals which are at temperatures considerably below that of their melting points, the amorphous phase is, as is illustrated in the author's Plate 3 (page 623), of such an extremely viscous nature as to be incapable of plastic deformation, but to possess very considerable tenacity or cohesion. If, therefore, we accept the author's theory that the intercrystalline fracture of his tungsten is due to the brittleness of the amorphous cement, then such a form of fracture should be the rule rather than the exception in other metals. The paper refers to one other case only, viz., iron at the temperature of liquid air; but the author does not state whether he has obtained direct experimental confirmation of the fact, nor does he quote any authority to support it. In Hadfield's paper (No. 38 in the bibliography), while it was shown that iron was brittle at this low temperature, yet I can find no reference as to whether the brittle fractures followed the crystal boundaries or the crystal cleavages. Since, however, the brittleness was accompanied by a high tenacity, I should prefer to assume the latter rather than the former.

When a metal tends to fracture around rather than through the crystals, it must surely be an indication that the *tenacity* of the cement is less than that of the crystals, and, provided the thickness of the cement is sufficiently small, its lack of plastic ductility does enter into the matter. All metals in their normal state tend to fracture around the crystals when at temperatures just below those at which they freeze (bibliography, No. 31). In this range, the amorphous cement is more capable of plastic deformation than the crystalline, but possesses considerably less tenacity. Below the temperatures which the author terms those of "equi-cohesion," the fractures change to cleavage. If, at still lower temperatures, the fractures again reverted to intercrystalline, then it would have to be assumed that the tenacity of the amorphous phase had begun to decrease with temperature, or at least to increase at a lower rate than the crystalline, and that the two curves in the author's Plate 3 again crossed one another. Such a reversion is most unlikely, and opposed to all our experimental knowledge.

We do, however, know of cases in which certain metals, which normally break through the crystals, may be so altered in character as to tend to break around them. Typical examples of this are:

(*a*) Gold containing small percentages of bismuth. It has been shown that the weakness is due to the presence of a fragile gold-bismuth eutectic.[5]

(*b*) Pure iron which has been annealed at certain temperatures and in certain atmospheres, and slowly cooled through a certain range. In this case there is evidence to indicate that the weakness is due to the formation of an iron-oxide eutectoid.[6]

(*c*) Nickel-chrome and certain other alloy steels, when hardened by quenching, annealed below the critical range, and then allowed slowly to cool through a range of temperature in the neighborhood of 500° C. In this case there is as yet no experimental evidence to indicate what is the important factor, but the general nature of the phenomena again points to the formation of a eutectoid.[7]

In the above examples it would appear that, contrary to the normal amorphous cement being responsible for the intercrystalline weakness, it is rather the fact that the presence of some foreign material between the crystals has interfered with its proper formation. It is suggested that it is to some similar interfering agent that the weakness found in tungsten, as made by the process now employed, should more properly be ascribed. Future researches will, I think, find a method of preparing metallic tungsten which shall be free from such weakness and have mechanical properties in line with those of other metals. The preparation of such material would be of great practical importance and would well repay the labor of a lengthy investigation.

It is fairly obvious that severe deformation by such processes as wire

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[5] J. O. Arnold and J. Jefferson: Influence of Small Quantities of Impurities on Gold and Copper. *Engineering* (Feb. 7, 1896) **61,** 176–179.

[6] J. C. W. Humfrey: The Intercrystalline Fracture of Iron and Steel. *Carnegie Scholarship Memoirs,* Iron and Steel Institute (1912) **4,** 80–105.

[7] H. P. Philpott: Some Experiments on Notched Bars. March, 1918, meeting, Institute of Automobile Engrs. Will probably be published in *Proceedings* (1918–19) **12.**

drawing or swaging will tend to strengthen a metal which originally lacks intercrystalline cohesion, since it must result in a certain amount of dovetailing of the crystals into one another. If, however, it is assumed that the original brittleness was due to the presence of the amorphous cement, it would appear that the contrary would be the case, since many additional planes of weakness would be produced by the formation of amorphous matter in the slip planes.

Cold working of a metal must necessarily, I think, be accompanied by the formation of internal stresses, partly due to unequal external loading, partly to the difference in resistance offered by the crystals and the amorphous matter surrounding them and formed during deformation, and partly to varying plasticities of different crystals, owing to the way they are oriented to the main axes of strain. When a certain degree of deformation has been given, the internal stresses at certain places may rise to that of the maximum tenacity of the metal. Further deformation at the same temperature must then immediately lead to internal rupture; this is the state of maximum tenacity which can be produced in the metal by cold work. If, however, the temperature of the metal be reduced, the tenacities of both the crystalline and the amorphous phases are increased, and further deformation may now be applied before the internal stresses again become equal to the tenacities, *i.e.*, the metal has acquired additional ductility. This explanation is applicable to all metals, including tungsten, and is independent of the relative tenacities of the crystalline and amorphous phases, provided only that both increase with falling temperature.

ZAY JEFFRIES (author's reply to discussion*).—Messrs. Merica's and Humfrey's criticisms of some of my conclusions are rendered in the true scientific spirit by means of which differences of opinion are gradually smoothed out and all participants are benefited. It is true that the statement of generalizations on such a complex subject is hazardous, but I shall endeavor to point out the explanations for the discrepancies noted. Owing to the fact that I am now submitting a long paper on the same general subject to the Institute, my present remarks will be brief.

Dr. Merica's Assumptions

I agree with No. 1 and No. 2, with the modification that the *A* (amorphous) metal between grains of annealed metal has substantially the same properties as that at slip planes in deformed metal, but the two have different arrangements with reference to *C* (crystalline) metal and they may be in different conditions as regards internal stress, thickness, and nature of contact with *C* metal.

Assumption 3.—I believe Fig. 1 is wrong. I know of no evidence to

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* Received Oct. 30, 1918.

substantiate the curves. I believe ϵ_C (elongation of C phase) $= 0$ (approximately) at all temperatures and ϵ_A (elongation of A phase) has finite values at elevated temperatures only; but these play no important direct role in the ordinary deformation of metals at low temperatures. I believe E_C (elastic limit of C phase) $= T_C$ (tensile strength of C phase) because as soon as C metal begins to deform permanently it would rupture were it not for the A metal generated by its breakdown. We know that $E_C = T_C$ in brittle crystals and a crystal cannot be permanently deformed without generation of A metal. Therefore Dr. Merica's T_C, in reality, equals $T_{(A+C)}$. Also $E_A = T_A$ for all practical purposes, because when A metal permanently deforms no new stronger phase is formed and hence any unit load which will permanently deform it will eventually break it. If the above is true, Dr. Merica's Fig. 2 is wrong, there being but one set of curves for E_A and E_C which correspond to my Plate 3. The duration of load is very important in A metal. The above reasoning should also be applied to assumptions 4 and 6.

Assumption 8b.—I believe this is not warranted from the evidence at hand and may lead to wrong conclusions.

Assumption 9.—I should like to add a fourth potential path of rupture, namely, at the contact between A and C metals.

Dr. Merica's Conclusions

(*a*) I have concluded, after examining considerable evidence, that at room temperature and lower, in equiaxed tungsten, there is internal stress between A and C metals, probably due to differences in their coefficients of thermal expansion. Call this internal stress IS and Dr. Merica's $T_A < E_C$ becomes T_A, or $E_A - IS < E_C$. My EC_E (equicohesive temperature for elastic limit) is not as Dr. Merica states; it is the temperature at which mixtures of A and C metals have the same resistance to deformation regardless of the initial quantity of A metal present. Obviously, when C metal or a mixture of A and C metals is deformed by Brinell impression, A metal is generated. If the total load is kept constant, the deformation gradually increases the area of contact of the ball and decreases the unit load until a point is reached at which substantially no further deformation takes place; this measures the resistance to deformation of mixtures of A and C metals. The same is true of my hairpin tests; the deformation caused a lessening of the load by shortening the lever arm. In all cases the deformation was caused, to my mind, largely by the movement at the slip planes and slightly, and more particularly at the higher temperatures, by actual deformation of A metal.

(*b*) Dr. Merica's condition for the increase in ductility at lowered temperatures is that the quantity $(T_A - E_C)$ must increase with decrease in temperature. In view of the assumption that $T_A = E_A$, Dr. Merica's condition is identical with mine. It seems to me that increase in ductility of a deformed metal by lowering the temperature has no relation to rupture at grain boundaries; the former holds for the range from highest temperature down to that of maximum ductility and the latter takes place only when the internal stresses between A and C metals exceed a certain amount. Briefly, the cohesion of A and C metals, the quantity and arrangement of the A metal and time of rupture (whether simultaneous or gradual) and rate of loading (especially at high temperatures) will determine the breaking load at a given temperature in the ductile temperature range. With other conditions constant, the breaking load will increase with decrease in temperature proportional to the cohesion of A metal. But with decrease in temperature the elastic limit of the deformed grains, which consist of A and C metals, must increase less than the elastic limit of A metal and more than the elastic limit of C metal. Hence the breaking load increases faster than the elastic limit of deformed grains and ductility must increase with decrease in temperature.

(*c*) Again Dr. Merica's discrepancy is explained by the existence of internal stress between A and C metals. It seems to me that Dr. Merica has unnecessarily complicated this subject by the assumption that the tensile strengths and elastic limits are not equal. With my assumptions, E_A is the average elastic limit of C metal in all directions and E_A is greatly affected by the duration of the load, Dr. Merica's Fig. 2 assumes that $T_A = E_A$ at the melting point. We are all familiar with the brittleness of amorphous substances at very low temperatures, which means that at these temperatures $T_A = E_A$ also. According to Fig. 2, E_A and T_A become farther apart as the temperature decreases and hence could never intersect. Also, Dr. Merica's T_C and E_C curves cannot intersect at low temperatures, yet we know at these temperatures they are equal. The obvious conclusion is that $T_A = E_A$ and $T_C = E_C$.

Reply to Mr. Humfrey

Mr. Humfrey first gives his conception of the quantity and arrangement of the A metal and states that I consider the amorphous cement as occupying a volume in the mass comparable with that of the crystals. My opinions on this subject coincide in the main with Mr. Humfrey's, and I have so stated in reference 34.

My previous remarks on internal stresses between A and C metals at low temperature apply to Mr. Humfrey's suggestions as to the nature of intercrystalline cohesion. He makes one statement, however, which, on its face, seems to be a contradiction to the whole hypothesis; namely, that if the A metal at the grain boundaries gives way for any cause at a load lower than the elastic limit of the C metal, the A metal at the slip planes in the deformed grains should do the same. It is a fact—and this will be discussed fully in my next paper—that the A metal at the slip planes does produce brittleness and, eventually, weakness in the deformed grains, but the effect of the deformation is to lower the temperature at which this brittleness and weakness first appear on cooling. The intercrystalline fracture occurs in recrystallized tungsten at 200° C. and lower. Below room temperature, equiaxed tungsten becomes weaker with decreasing temperature; also, severely worked tungsten is weaker in liquid air than it is at room temperature, and it is also brittle. The above indicates that either the path of rupture along the amorphous slip planes in deformed grains is greater than at grain boundaries in equiaxed tungsten, or that the *A* metal in deformed grains which has been generated at a comparatively low temperature must be cooled to a lower temperature than that at the grain boundaries to produce a given amount of internal stress between A and C metals.

Mr. Humfrey's belief that future researches will find a method of preparing metallic tungsten which will be free from intercrystalline weakness has been shared by many workers on tungsten. Although recognizing that such a thing may be possible, I am of the opinion that fine-grained equiaxed tungsten will never be produced in such a form that it will be ductile at ordinary temperatures like other metals. Lamp filaments recrystallized in a vacuum and heated at high temperatures for over 1000 hr. are not ductile at ordinary temperature unless a single grain occupies the whole cross-section of the filament for a considerable length, or the grains are greatly elongated, as indicated in Fig. 49. Tungsten rods composed of large grains can be worked and recrystallized in such a manner that each large grain gives birth to many small equiaxed grains which show intercrystalline fracture at room temperature. If we assume that the original large grain should have concentrated its impurities at its own boundary, the small grains produced from it should be cemented together with substantially pure *A* metal. Again, the *A* metal at the slip planes in deformed tungsten grains behaves like the intercrystalline material when a sufficiently low temperature is reached.

I am unable to follow Mr. Humfrey's reasoning with reference to the increase in ductility in a strain-hardened metal with decrease in temperature. It is true that an increased tenacity due to release of strains increases ductility at a given temperature, but when the increased tenacity is gained by lowering the temperature, it must be remembered that the resistance to deformation increases also. What makes the tenacity increase faster than the resistance to deformation, Mr. Humfrey's explanation does not say. My contention that in ductile metals the amorphous metal controls the tenacity and that its cohesion increases with decrease in temperature faster than the crystalline metal, satisfies the above conditions, it seems to me, and furthermore, this generality holds for all metals.