code 1

TT65-64816

624909

FTD-TT-65-1010

TRANSLATION

RECRYSTALLIZATION OF HIGH-MELTING METALS IN WELDING AND SOLDERING

By

N. V. Glebtsev and G. N. Klebanov

FOREIGN TECHNOLOGY
DIVISION

AIR FORCE SYSTEMS COMMAND

WRIGHT-PATTERSON AIR FORCE BASE
OHIO

UNEDITED ROUGH DRAFT TRANSLATION

RECRYSTALLIZATION OF HIGH-MELTING METALS IN WELDING AND SOLDERING

BY: N. V. Grebtsev and G. N. Klebanov

English pages: 16

SOURCE: AN SSSR. Izvestiya. Otdeleniye Tekhnicheskikh Nauk. Metallurgiya i Toplivo. (Russian), No. 5, 1961, pp. 62-69.

S/0180-061-000-005

TP5001635

	PREPARED BY: TRANSLATION DIVISION FOREIGN TECHNOLOGY DIVISION WP-AFB, OHIO.

Date 9 Nov. 19 65

S

RECRYSTALLIZATION OF HIGH-MELTING METALS IN WELDING AND SOLDERING*

N.V. Grebtsev and G.N. Klebanov

(Moscow)

The basic problem encountered in welding high-melting metals consists in improving the mechanical properties of the welded joints, and their plasticity in particular. To develop methods for joining these metals with themselves and other metals, it is necessary to have available the characteristics of the metal's recrystallization processes under the conditions of the heat cycles of welding and soldering.

Data on the influence of the heat effect in welding and soldering on changes in the structure and mechanical properties of the basic metal enable us to determine the range of optimum conditions for the various methods of welding them by fusion and in the solid phase, and to select effecient methods and conditions for soldering.

In recent published material on the processes of recrystallization in high-melting metals, there is no information at all concerning their behavior under the conditions of the heat cycles encountered in welding and soldering. Literature data on the recrystallization temperatures of high-melting metals were obtained during heating in furnaces or rapid conduction-current heating with subsequent prolonged holding at the specified constant temperature. Nor are there any data on the influence of the basic parameters of the heat cycles on the metal's recrystallization processes in the zone of the heat effect.

The present study was concerned with recrystallization processes in welding and soldering of molybdenum, tungsten, tantalum and niobium and the influence of these processes on the structure and mechanical properties of these metals in the zone around the seam.

Depending on the methods and conditions of welding or soldering, one of the basic parameters of the heat cycle and a determining one for the recrystallization process – the rate of heating – may vary over an extremely wide range. It would be of interest to study the influence of heating rate on the temperature of onset of working recrystallization and that of the time for which the metal is above the recrystallization temperature on grain growth (cumulative recrystallization). In welding and soldering, it is precisely these two parameters of the heat cycle that are subject to regulation.

The maximum temperature to which the zone around the weld is heated, another factor that influences grain size, is determined by the method used in forming the joint. In fusion welding, it is near the melting point of the basic metal, while in solid-phase welding it is not, as a rule, below the recrystallization temperature; in soldering it is close to the melting point of the solder.

The influence of heating rate on the initial temperature of recrystallization was studied by the IMYeT-1 method [1], using specimens whose sections varied over their length and which were heated at different rates arrived at by varying the conduction current. The heating rate of a section passed through at a distance of 5 or 10 mm from the central waist of the specimen was varied from 40 to 2200 deg/sec.

The heating rates used in the experiments covered the range of heating rates actually encountered in the various modes of welding and soldering. In the case of welding, they lay in the range from 2000 to 2500 deg/sec (for metal 1.0-1.5 mm thick), and in soldering they were no more than a few hundred degrees per second.

After heating and cooling, the specimens were studied metallographically. The zone of onset of recrystallization was identified from the appearance of the first equiaxial-grain nuclei in the rolling texture. Knowing the distributions of temperature and heating rate along the specimen, we could determine the heating rate in this zone and the maximum temperature, for which we would take the temperature of initial recrystallization at the heating rate in question.

The temperature interval bounding the zone of onset of working recrystallization (appearance of the first equiaxial-grain nuclei in the texture) and its end was determined for niobium not only metallographically, but also from the change in microhardness.

These same specimens were used to determine the influence of the time for which the metal was held above the recrystallization temperature (at various maximum temperatures) on grain size and the influence of grain size on hardness.

The specimens were heated in an argon-filled airtight chamber. In the mechanical tests in the IMYeT-1 unit, the specimens were protected from oxidation by blowing argon through the nozzle.

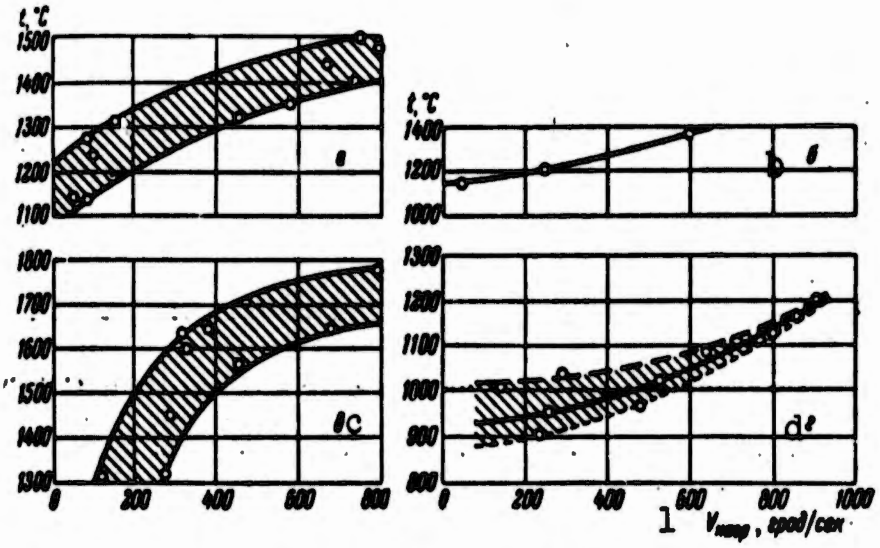

Fig. 1. Influence of heating rate on temperature of onset of crystallization in molybdenum (a), tantalum (b), tungsten (c) and niobium (d); the crosshatched region on diagrams a, b and c corresponds to the scatter of the temperature values obtained. 1) V_{nagr}, degrees/sec.

The temperatures at points in the center part of the specimen were measured with a tungsten-rhenium (W + 5% Re; W + 20% Re), and those in more remote regions with platinum-platinum-rhodium or chromel-alumel thermocouples; they were registered with an N-700 magnetoelectric oscillograph. The thermocouple junction was welded by capacitor discharge into a hole drilled in the specimen.

The experiments to determine the influence of heating rate on the temperature at which recrystallization begins in molybdenum and tungsten established that this temperature becomes higher in more rapid heating (Fig. 1, a, c). At comparatively moderate heating rates (50 degrees//sec for molybdenum and 200 for tungsten), it is 1200 and 1300°, respectively; with an increase in heating rate to 800 deg/sec, the onset temperatures of recrystallization rise to 1450 and 1700°, respectively. However, this increase does not continue without limit. When tungsten is heated faster than 500 deg/sec and molybdenum faster than 800 deg/sec, the onset temperature of recrystallization tends to a certain limiting value.

Since the maximum heating rates in the experiments were close to those of arc welding, and the specimens were cooled in water (practically instantaneously), it may be assumed that it is impossible to avoid the appearance of a recrystallized zone in any of the methods used to weld molybdenum and tungsten by fusion.

According to [2, 3], recrystallization of technical tantalum begins at 1200° and is complete at 1800°. As the degree of deformation of tantalum varies from 2.5 to 90%, the initial temperature of recrystallization drops from 1300 to 1200°, reaching 1250° at 50% deformation [4].

In welded constructions, as a rule, sheet tantalum or tubular billets with degrees of deformation ranging from 70 to 90% are employed. It may be assumed that working recrystallization begins at 1200° under conditions of relatively slow heating and subsequent long holding times for such metal.

Obviously, the very high heating rates encountered in welding should also influence the temperature of recrystallization onset in tantalum. Studies carried out on variable-section specimens indicated that as the heating rate varies from 100 to 600 deg/sec, the initial temperature of recrystallization rises from 1150 to 1270° (Fig. 1b). In the range of heating-rate variation studied, tantalum, unlike molybdenum and tungsten, showed no tendency to stabilization of the recrystallization temperature in rapid heating. It may be assumed that as the heating rate is further increased, this temperature will also tend to some limiting value.

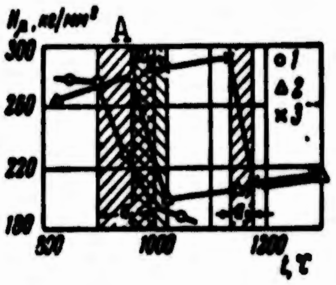

Fig. 2. Change in temperature intervals of recrystallization (a_1, a_2, a_3) during heating of niobium at various rates, as follows in deg/sec: 1) 250-300; 2) 460-520; 3) 800-880; $a_1 > a_2 > a_3$. A) H_μ, kg/mm^2.

The low recrystallization-onset temperature (1150°) at low heating rates can be accounted for in terms of the use of purer tantalum than was the case in earlier studies. As we know, the degree of contamination of tantalum and niobium with impurities – first and foremost, oxygen and carbon – influences their recrystallization-onset temperatures to a significant degree.

We established that as the heating rate of niobium is varied from 280 to 860 deg/sec, its initial recrystallization temperature rises from 950 to 1175° (Fig. 1d). On the diagram of Fig. 1g, the middle solid line corresponds to the temperatures at which equiaxial-grain nuclei readily distinguishable under the microscope form in the rolling texture. After the niobium has been heated to the temperatures indicated by the points on the lower curve and then cooled, its hardness shows a sharp decrease. This is associated with recovery processes and the onset of working recrystallization.

The upper broken curve in Fig. 1g corresponds to the temperatures at which working recrystallization is practically complete throughout the entire volume of the metal, at which point grain growth begins. Here the hardness is observed to stabilize.

The temperature ranges of recrystallization as measured through the hardness change of the specimens after heating at various rates are shown in Fig. 2.

With increasing heating rate, the temperature interval between the onset and completion of working recrystallization becomes markedly narrower as a result of a rise in the temperature of recrystallization onset. Here the structure of the metal at the end of recrystallization is found to be finer-grained. This phenomenon is accounted for as follows. The final grain size after recrystallization is determined by the ratio of the rates of two processes: new equiaxial-granule nucleation and the growth of these granules. Usually, the growth rate is higher than the nucleation rate, since the activation energy of nucleation is greater than that of growth. In rapid heating, however, the difference between the rates at which these processes advance becomes less marked.

New equiaxial-grain nucleation takes place in those regions of the metal where the crystal lattice has been most severely distorted as a result of deformation. Consequently, the greater the deformation of the metal and the larger the type II and III stresses in it, the greater the number of nuclei that arises per unit of time. As the metal is heated, the recrystallization process is preceded by a recovery or relaxation process in which some of the lattice distortions due to the stresses are relieved.

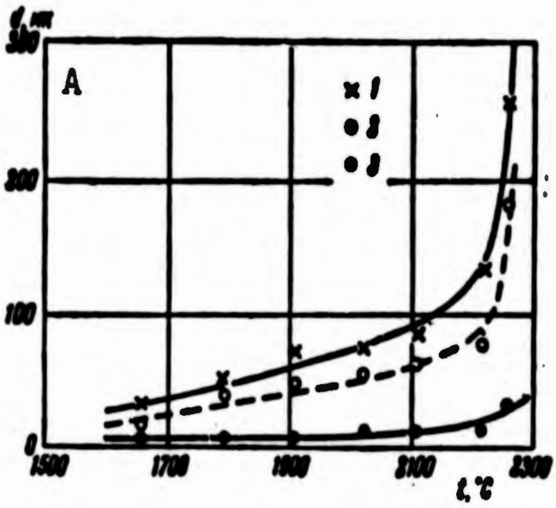

Fig. 3. Growth kinetics of molybdenum grain in heating: 1) Coarse grains; 2) medium; 3) fine. A) d, μ.

The number of recrystallization nuclei and the nature of the metal's vital structure depend on the duration of the recovery processes, i.e., in the last analysis on the heating rate: the shorter this time or the higher the heating rate to the recrystallization temperature, the less complete will be the relaxation processes and the finer should be the grain after working recrystallization has been completed.

Thus, grain size after working recrystallization is determined by the number of nuclei formed under the given conditions in the temperature range at which recrystallization begins: the greater the number of nuclei, the smaller the final dimensions of the grain.

In studying the kinetics of grain growth (cumulative recrystallization) in molybdenum and tantalum during the welding heat cycle, flat rod specimens were heated and cooled on a predetermined cycle. When the required temperature had been reached, the specimens were cooled abruptly in water.

It was established by metallographic analysis that under the conditions of rapid heating, molybdenum shows marked grain growth (Fig. 3) at temperatures above 1600γ. Above this temperature, together with new equiaxial-grain nucleation, we observe the growth of grains that had formed earlier. Their sizes increase by factors of approximately 2-4. At temperatures above 2100°, the growth rate increases sharply, with the area occupied by fine-grained regions diminishing and the transverse size of the coarse grains reaching 250-300 μ at 2200-2250°.

The grain growth of tantalum does not terminate when heating does, but continues even during cooling, although at a dimishing rate. On heating to 1200, the structure of tantalum still retains elements of the rolling texture (well-defined orientation of individual grain groups). Working recrystallization is complete at 1400° and cumulative recrystallization begins. As the temperature rises to 1800°, the grains reach considerable size (105 μ), and on cooling from this temperature to 1625°, they reach the practical maximum (120 μ); on further cooling, the increase in average grain diameter is insignificant (less than 132 μ).

The microhardness of these specimens was measured after cooling. Rapid grain growth during heating is accompanied by a considerable drop in the microhardness.

To evaluate the ultimate changes in the structure and mechanical properties of molybdenum, tungsten, tantalum and niobium in the zone of the weld heat effect, specimens were heated and cooled in accordance with predetermined heat cycles.

The results of these studies enable us to conclude that the most rapid molybdenum and tungsten grain growth is observed with short holding times above the recrystallization temperature (Fig. 4): for tungsten less than 1 sec at a maximum temperature of 1700°, less than 1.5 sec at 1800°, less than 2 sec for 1900°; for molybdenum, less than 2 sec at a maximum temperature of 1400°, less than 4 sec at 1600° and less than 7 sec at 1800°.

These data indicate that shortening the heating time is of value only for welding conditions under which the metal is heated to temperatures near the melting point. In soldering with working temperatures in

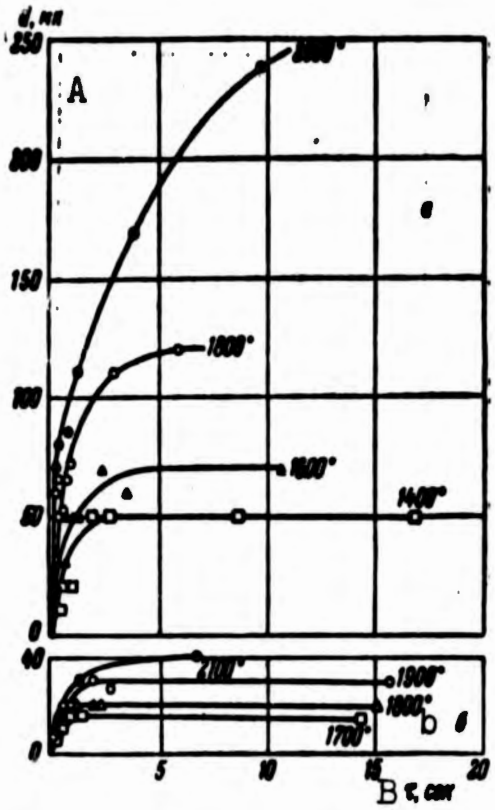

Fig. 4. Influence of maximum heating temperature with various holding times above recrystallization temperature (τ, sec) on average grain size (d, μ) in molybdenum (a) and tungsten (b). A) d, μ; B) τ, sec.

the 1600-2000° range, the duration of the process has little influence on the grain size of the ground metal. At higher working temperatures, the duration of the soldering process should be shortened, for example, by heating with high-frequency currents.

Among all of the parameters of the heat cycle in the ranges in which they vary in actual welding regimes, heating temperature exerts the strongest influence on the growth processes of technical tantalum grain. When specimens are heated to 1600-2300° (heating time to t_{max} 2.0 sec, cooling at a rate of 330-430 deg/sec), the average grain size increases from 10 to 160 μ.

Variation of the heating rate exerts less influence on the ultimate grain dimensions over almost the entire zone of temperatures studied; for example, as the heating rate is reduced from 500 to 100 deg/ /sec, the grains grow by factors of approximately 1.2-2.0 (t_{max} = 2120°, cooling rate 335 deg/sec).

Variation of the metal's cooling rate exerts the least influence on the final grain size. When the cooling rate is increased from 100 to 400 deg/sec, the final grain size diminishes by a factor of 2 if the metal has been heated to 1600°, by a factor of 1.5 for 1200° and by a factor of 1.2 for 2360°. This indicates that the influence of cooling rate on grain size becomes weaker as the maximum heating temperature is raised.

Like tantalum, niobium is inclined to significant grain growth on heating to temperatures above the recrystallization temperature; however, the influence of the individual welding heat-cycle parameters on the final grain dimensions is somewhat different for this metal.

This difference consists, firstly, in the fact that under certain conditions (rapid heating and very fast cooling), the grain size in niobium is smaller at temperatures above 2100° than for tantalum. Secondly, the difference in the influence of heating and cooling rates is smaller than tantalum, all other conditions be same. The absolute influence of these parameters comes most distinctly into evidence at heating and cooling rates between 200 deg/sec, i.e., under the conditions of soldering heat cycles. Thirdly, grain size diminishes markedly as the cooling rate is increased (Fig. 5).

The manner of cooling affects the final grain size very strongly, particularly in the region of high temperatures (2120°), where it is possible, by abrupt cooling (445 deg/sec) of specimens that have been heated slowly (190 deg/sec) to obtain a finer grain than in specimens heated at high speed (1200 deg/sec) but cooled slowly (24 deg/sec).

Data on the influence of the individual parameters of the welding and soldering heat cycles on the final grain size in tantalum and niobium are generalized in the form of recrystallization diagrams (Fig. 6) for the heat-cycle conditions of welding and soldering.

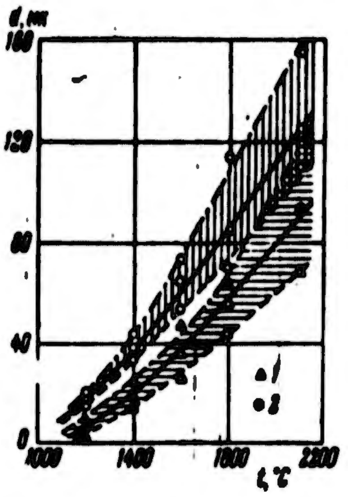

Fig. 5. Change in niobium grain size at various temperatures as a function of manner of heating and cooling: 1) Metal held above recrystallization temperature for 0.6-1.4 sec; 2) for 2.7-6.5 sec; the dot-dash line represents slow cooling and the dashed line abrupt cooling. A) d, μ.

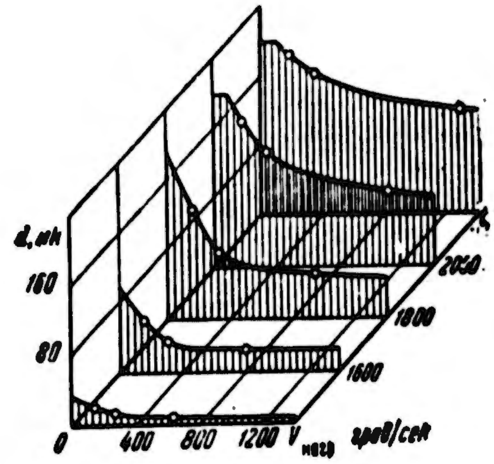

Fig. 6. Recrystallization diagram for niobium under conditions of welding and soldering. A) d, μ; B) V_{nagr}, degrees/sec.

In contrast to the recrystallization diagrams for isothermal conditions, these diagrams have been constructed using heating (cooling) rate, temperature and average grain size as coordinates. The diagrams have been constructed for metal deformed to 70-90%, on the assumption that variation of the degree of deformation within this range does not markedly influence grain size up to temperatures of the order of 2600° for tantalum and 2000° for niobium [4].

In the zone of variation of maximum temperature, heating rate and cooling rate corresponding to the soldering heat cycles, the influence of these parameters on the grain size in tantalum is not striking. Thus, a change in the maximum heating temperature corresponding to the soldering working temperature from 1640 to 2000° results in an increase in average grain diameter from 25 to 100 μ, while reducing the rate of heating to 1640° from 100 to 10 deg/sec increases it from 25 to 30-35 μ. Under the conditions of welding heat cycles, which are characterized by high maximum temperatures ranging up to the melting point, the influence of heating rate comes strongly into evidence. This permits us the conclusion that in selecting a method and conditions for welding tantalum less than 2 mm thick, the basic parameter of the heat cycle that should be regulated to limit grain growth in the zone around the weld is the heating rate or the time spent by the metal above the recrystallization temperature during heating.

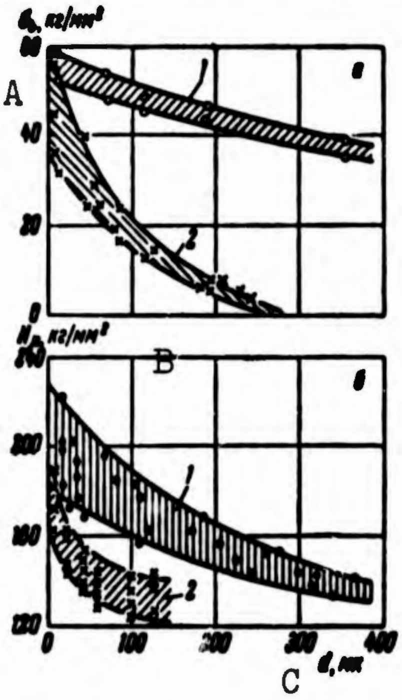

Fig. 7. Strength (a) and hardness (b) of tantalum (1) and niobium (2) as a function of grain size. A) σ_b, kg/mm^2; B) H_μ, kg/mm^2; C) d, μ.

In soldering of niobium, on the other hand, the rate of heating to the working temperatures has a marked influence on the final grain size. For example, at a soldering temperature of 1600°, a change in heating rate from 50 to 200 deg/sec reduces the average grain diameter from 85 to 50 μ, or from 170 to 115 μ at 1300°. At higher soldering temperatures, this influence is less pronounced.

Under the conditions of niobium welding, only the heating temperature is of decisive importance, with the grain in this case growing approximately to the same size as in tantalum welding (about 150 μ on heating to 2200° at a rate of 600 deg/sec). However, due to the higher temperatures to which the weld zone is heated in welding tantalum, the final grain size in this metal is 40-50 μ larger than in niobium.

Cooling rate exerts a marked influence on the final grain size in niobium only in the high-temperature region; for example, as the cooling rate is varied from 100 to 600 deg/sec after heating to 2100 and

1600°, the grain dimensions decrease from 110 to 70 and from 35 to 25 μ, respectively.

At the present time, the literature presents no specific information on the influence of grain size on the mechanical properties of tantalum and niobium. However, it has been demonstrated on other metals [5] that this effect may be quite considerable.

The relationship between the strength and hardness of technical tantalum and niobium and the grain size was determined in this study on specimens that had been heated to various maximum temperatures. After such treatment, the specimens were tensile-tested at room temperature (Fig. 7). For tantalum, an increase in average grain diameter from 20 to 350 μ results in a decrease in ultimate strength from 52-58 to 37-39 kg/mm^2 and a decrease in microhardness from 180-220 to 140-142 kg/mm^2. With increasing grain size, the scatter of the mechanical-property data, and that of hardness in particular, diminishes noticeably.

For niobium, strength drops from 35-55 to 5-6 kg/mm^2 when the grain size increases from 10 to 240 μ; the hardness diminishes from 160-190 to 120-145 kg/mm^2 as the grain is enlarged from 5 to 120 μ, with the sharpest drop in hardness taking place in the 5-to-40-μ grain-size interval; as the grain size increases further, we observe a stabilization of hardness.

Data on the influence of the individual welding and soldering heat-cycle parameters on the recrystallization and grain-growth processes in tungsten, molybdenum, tantalum and niobium enable us to conclude that in welding these metals it will obviously not be possible to prevent the appearance of a region with very coarse grain in the zone of the weld heat effect – a region that possesses low mechanical properties as a result. For tantalum and niobium, we should expect a decrease in the strength of the welded joint by 32-40% with respect to the basic metal.

To reduce the extent of coarse-grained zones in welding up tantalum structures, welding should be conducted with small energies per unit length (with high currents and high welding speeds), or the heat sources used should ensure deep penetration and concentrated heating, as does the electron beam. Since tantalum is more strongly inclined to grain enlargement than is niobium, the cooling rate exerts a weaker influence on the final grain dimensions in the zone around the welded joint in tantalum. Hence no special measures are required to regulate cooling conditions in welding tantalum.

Everything said concerning sharp concentrated heating in welding tantalum applies in full to niobium as well. In contrast to the case of tantalum, it is helpful in welding niobium to take supplementary measures that contribute to more rapid cooling of the welded joint.

Niobium and niobium-based alloys should be welded at higher-than-usual speeds, using various heat-dissipating expedients in the process (cooled backing strips, copper overlays, and the like).

Conclusion. In the rapid heating encountered in welding, the initial recrystallization temperatures of molybdenum and tungsten rise by 250 and 400°, respectively, tending toward some limiting value. For tantalum and niobium, this temperature rise amounts to 120-150 and 170-200°.

Under the conditions of rapid heating, considerable grain growth is observed in molybdenum at temperatures above 1600°. Cumulative recrystallization proceeds most rapidly as the heating temperature is increased. As the molybdenum is held for longer times above the recrystallization temperature during heating, grain growth is observed when this holding phase is short. Hence it is of value to shorten the duration of heating only for welding conditions under which the basic metal is heated to temperatures near the melting point. In soldering with working temperatures below 1600°, the duration of the process has little effect on the grain size of the ground metal. At higher soldering temperatures, it is advisable to heat with high-frequency currents.

Among all of the parameters of the welding heat cycle, that exerting the strongest influence on the processes of grain growth in tantalum and niobium is again the heating temperature, followed in order of importance by the holding times above the recrystallization temperature in heating and cooling. Hence these metals must be welded with heavy currents and at high speeds, or sources that ensure highly concentrated heating and deep penetration (electron beam) should be used.

The recrystallization diagrams constructed for tantalum and niobium on the basis of these studies for heating conditions approximating those of welding and soldering enable us to specify optimum working parameters that will ensure, for a given welding or soldering process, minimum grain size in the zone of the heat effect and the smallest possible size of this zone.

Received
27 June 1961

REFERENCES

1. Shorshorov, M.Kh., Klebanov, G.N. Metodika i mashina IMYet-1 dlya issledovaniya izmeneniy struktury i mekhanicheskikh svoystv metalla pri svarke [IMYeT-1 Process and Machine for Studying Changes in the Structure and Mechanical Properties of Metal During Welding], Filial VINITI AN SSSR, Peredovoy proizvodstvennyy i nauchno-tekhnicheskiy opyt [Academy of Sciences USSR All-Union Institute of Scientific and Technical Information Branch, Advanced Production and Scientific-Technical Experience], 1957, No. M-57-134/12.

2. Mayers, R. Nekotoryye svoystva tantala [Certain Properties of Tantalum], Collection entitled. Niobiy i Tantal [Niobium and Tantalum], Part 1, IL [Foreign Literature Press], 1954.

3. Uensh, G., Brukkart, K., Deybler, R. Rekristallizatsiya tantala [Recrystallization of Tantalum], Collection entitled. Niobiy i tantal, Part 1, IL, 1954.

4. Savitskiy, Ye.M. Redkiye metally i splavy [Rare Metals and

Alloys]. Dom tekhniki [House of Engineering], Moscow, 1959.

5. Sabitskiy, Ye.M. Vliyaniye temperatury na mekhanicheskiye svoystva metallov i splavov [Influence of Temperature on the Mechanical Properties of Metals and Alloys]. Izd-vo AN SSSR [Academy of Sciences USSR Press], 1957.

Manuscript Page No.	[Footnotes]
1	The work was done in the Welding Processes Theory Laboratory of the A.A. Baykov Metallurgical Institute under the supervision of Corresponding Member of the USSR Academy of Sciences N.N. Rykalin.

Manuscript Page No.

[Transliterated Symbols]

= IMYeT = Institut metallurgii = Metallurgical Institute

= nagr = nagrev = heating