FOREWORD

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This report covers work conducted from October 1, 1960 to October 31, 1961.

ABSTRACT

Studies have been made of (a) the strain-aging behaviour and (b) the effect of pre-straining and high temperature annealing on molybdenum crystals of two different purity levels prepared by electron beam melting. One batch of crystals contained an average of 20 ppm total interstitial impurities, the other an average of 250 ppm interstitials with the major constituent being carbon.

Yield point experiments revealed no strain-aging in either material in the "as-grown" condition, but after a high temperature anneal followed by rapid cooling the material containing carbon showed appreciable aging effects, and the purer material weak effects. The results are explained in terms of the low solid solubility of the interstitial elements in molybdenum under equilibrium conditions at moderate temperatures.

The second series of experiments showed that pre-straining and annealing treatments which produce a sub-structure in molybdenum, also result in a strengthening of the material. The strengthening increases with increasing pre-strain and with increasing carbon content. The results indicate that sub-grain boundaries strengthen molybdenum in a qualitatively similar way to ordinary grain boundaries.

PUBLICATION REVIEW

This report has been reviewed and is approved.

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IMPURITY ATOM-DISLOCATION INTERACTIONS AND SUBSEQUENT EFFECTS ON MECHANICAL PROPERTIES OF REFRACTORY METALS

I. INTRODUCTION:

The refractory metals have in recent years been receiving ever increasing attention because their very high melting points indicate that they may find useful application as high temperature structural materials. As a consequence much effort is being currently expended in evaluating and attempting to improve the mechanical properties of these materials, such that their potentialities may be fully realized. Alloying is one possible method by which high temperature strength may be optimized; the use of mechanico-thermal treatments on the pure, or nearly pure, materials is another. The work described in this report is, in its general scope, directed toward an evaluation and understanding of one possible means of enhancing strength by the latter method; specifically, by the introduction of a suitable sub-structure, in this case into molybdenum.

General studies of the influence of sub-structure on mechanical properties have been rather sparse, but the work that has been reported (1-6)* does indicate that sub-grains can produce strengthening effects. In experiments with face-centred-cubic metals, for example, it has been shown (5) that the room temperature yield strength of nickel and nickel-titanium alloys increases with increasing sub-grain density. The introduction of a sub-structure into these materials (by pre-straining and annealing) also results in an appreciable reduction in the initial creep rate at 700°C (6).

On the basis of the above experiments it is to be anticipated that suitable sub-grain configurations should also strengthen the body-centred-cubic refractory metals. With these metals, however, complexities may be expected. Their mechanical properties are markedly affected by the small amounts of interstitial impurities which they almost invariably contain, so that the interaction between sub-grain boundaries and impurities has additionally to be considered. Sub-grain boundaries are, in effect, only arrays of dislocations and it is well known that strong interactions can occur between dislocations and interstitial impurities in the body-centred-cubic lattice. The importance of impurity - sub-boundary interactions takes on an added practical significance: by adding suitable impurities it may be possible to stabilize optimum sub-boundary configurations against the sub-grain coarsening that is known to occur under prolonged high temperature service conditions.

With the above factors in mind, the direct aims of the experi-

*Numbers in parenthesis indicate References.

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ments described here were two-fold. Firstly, to evaluate how effectively the individual interstitial elements carbon, oxygen and nitrogen can lock dislocations in molybdenum. Yield-point studies were used in this phase of the work, interest being confined at this stage to the behaviour of pure molybdenum, and that containing small amounts of carbon. Secondly, to study the effect of pre-straining and annealing on the subsequent yield strength of pure molybdenum and of the molybdenum-carbon "alloys".

II. EXPERIMENTAL:

A. Preparation of Materials.

It is a general characteristic of the body-centred-cubic transition metals that their mechanical properties are strongly influenced by the presence of minute quantities of interstitial impurities. As a consequence ultra-high purity materials are required for systematic studies of impurity effects in these materials. The best technique currently available for obtaining the required high degree of purification is electron-beam melting. This technique was used in the present work.

The electron-beam melting apparatus was of the floating-zone type, and has been described in detail elsewhere (7). In it, it is possible to process rods of up to 1/4" dia. in lengths of up to 9", at an operating vacuum of about 10^{-5} mm.Hg. The zone melting of molybdenum invariably resulted in the production of single crystals, an added advantage for the present experiments, preventing the interference of grain-boundary embrittlement in the strain-aging studies, and the complication of varying grain-size in the polygonization experiments.

Experience gained previously (7) indicated that the purification achieved during the electron-beam melting of molybdenum is a rather complex function of the purity of the starting material. The melting of Wah-Chang sintered molybdenum of initially high oxygen content results in good purification with respect to carbon, oxygen and nitrogen, but it is difficult to process large lengths of this material because excessive sparking causes coating of the emission filament. Climax arc-cast molybdenum of initially high carbon and low oxygen content can, on the other hand, be run very easily. However, although most of the gases are removed the carbon content is reduced only slightly after melting. To achieve the best overall purification, a starting material of balanced oxygen and carbon content (about twice as much oxygen as carbon) is needed. At the same time if the gas content is too high the material cannot be easily processed by ordinary floating-zone electron-beam methods.

At the commencement of the present programme an attempt was made to obtain a starting grade of molybdenum which could be zone-melted satisfactorily, and which would provide a finished product of high purity. Sintered material was obtained from all the major suppliers (Climax, Molybdenum Co., General Electric Co., Fansteel

and Sylvania) for this work, and each material was processed under conditions (single pass at zoning speed for 3"/hr. and a vacuum of 10^{-5} mm.Hg.) similar to those used for the Wah-Chang and Climax arc-cast stock. To avoid expense, material wastage and delays, no chemical analyses were obtained for these preliminary experiments; rather, yield strengths in tensile tests were taken as an indication of the purifications achieved.

All of the materials showed sparking during processing, but this was less violent than that for the Wah-Chang sintered specimens and did not prevent the completion of any of the runs. Upon tensile testing, however, all of the finished crystals showed yield strengths considerably in excess of those obtained from processed Wah-Chang stock, and occasionally sharp initial yield points were observed. This was taken as sufficient indication that these crystals would be of less use as ultra-pure reference material than those produced from Wah-Chang material. Attention was therefore diverted to methods of avoiding excessive filament coating whilst melting this very gassy material.

For normal running, the emitter in the electron-beam apparatus, a single turn tungsten filament, is located symmetrically inside a cylindrical focussing shield. The vertical rod specimen passes through central holes in the end closures of the shield. (Fig. 1(a)). With this arrangement the molten zone forms at a point on the specimen immediately opposite the filament, at a distance of about 1/4" from the latter, and the filament is directly exposed to sparks ejected from the zone.

The solution to the filament coating problem was derived from the observation that very few sparks are ejected from the molten zone other than in a direction rectilinear to the centre of the zone axis. This observation led to a realization that a slight deflection of the electron-beam away from the plane of the filament would provide protection from excessive coating. To achieve the deflection several different focussing shield arrangements were tried, the most successful being that shown in Fig. 1(b). Here a small truncated cone replaces the normally flat, bottom closure of the shield, the vertical height of which is increased slightly (by approximately the height of the cone). With this focussing shield arrangement the molten zone forms with its centre about 1/8" above the plane of the filament, and this provided sufficient filament protection to allow complete 9" runs on Wah-Chang material. Such material was always subsequently processed using the modified shield arrangement.

For the strain-aging and polygonization experiments, a batch of crystals was prepared from 1/8" dia. Wah-Chang and Climax arc-cast stock. Whenever possible, zone melting was carried out over the maximum 9" length of rod that the apparatus would allow. With

the arc-cast material, a single melting pass at a speed of 3"/hr. was found sufficient to remove almost all of the interstitial gases, leaving an appreciable amount of carbon. Two melting passes were used for the Wah-Chang material, thus enhancing purification. Little improvement in purity was found when the number of passes was increased beyond two. Typical analysis figures are given in Table I., and Fig. 2 shows the orientations (determined from X-ray laue back-reflection photographs) of the crystals used in the tests. Variations in the purity levels of the crystals prepared from a given stock material were apparently caused by the variations in the purity of the stock which, particularly for the arc-cast material, were quite large. The crystals used in critical experiments were individually analysed after testing.

B. Tensile Testing.

Tensile specimens were prepared from 2" lengths of crystal cut from the "as-grown" rods with a high speed, water cooled abrasive disc. Each crystal was electro-machined in a special jig to provide a reduced centre gauge section. The jig consisted of a 2" dia., 1.1" wide stainless-steel wheel (with rounded carriers) as the cathode, and a pair of co-axial pin vices to hold the specimen (the anode) parallel and close to the surface of the wheel. Both specimen and cathode were continuously rotated during machining, the lower part of the latter being immersed in 10% aqueous sodium hydroxide solution which formed by surface tension, a thin film bridging the gap between the electrodes when rotation was commenced. With a current-density of 2.32 amp. $in.^{-2}$ over the entire specimen, gauge sections 0.9" long by 0.07" ($\pm$ .001") dia., terminating in smoothly contoured shoulders, were produced in about 1-1/2 hrs. To improve their surface finish all of the crystals were given a light electro-polish in a methyl alcohol-sulphuric acid bath immediately before testing.

Tensile tests were made in a hard-beam, autographically recording machine of the same basic type as that described previously by Adams (8), at a constant cross-head speed of 0.02725 in./min. The crystals were gripped in the serrated jaws of carefully aligned pin vices attached to stainless-steel stems. For the strain-aging experiments the specially designed furnace assembly of Fig.3 was used. Here, the specimen A, attached to its grips B and C, was connected to the tensile machine members D and E by a water-cooled adaptor F at the top end and by a demountable water-cooled ball joint G at the bottom. A close-fitting clear silica tube furnace H enclosed the specimen which was sealed from the air by rubber gaskets I and J. Purified argon was passed through the closed system via an entry tube K and exit hole L, and a thermocouple M. sealed into the ball joint, was used to control temperature. Specimen temperatures of up to 850°C could be rapidly

(maximum of about 10 mins.) reached with this furnace, and at worst the gradient along the gauge length was less than 4°C. More important, annealing could be carried out with the specimen held aligned in the machine, an essential requirement when yield point studies are used for assessing strain-aging behaviour of a material (9).

For the polygonization experiments tensile specimens were prestrained by varying amounts at room temperature, and were then annealed (wrapped in molybdenum foil and away from the tensile machine) in an atmosphere of purified argon for 2 hrs.at 1500°C. This technique was similar to that used previously (7) for producing a sub-structure in molybdenum. Where the tests were comparative ones, the several specimens of a series were annealed together. After annealing, the yield strengths of the crystals were redetermined from room temperature tensile tests.

III. RESULTS:

A. Yield Points in Molybdenum Crystals.

It is appropriate at this stage to recall the experimental procedures that must be adopted if yield point studies are to provide an unequivocal indication of the ability of a material to strain-age. In the first instance it is essential that a "hard" tensile machine be used in order to show discontinuous yielding as a yield drop on a stress-strain curve. With "soft" machines discontinuous yielding appears as merely an extension under constant load, and this can be confused with the yielding behaviour of a non strain-aging material that has a low work-hardening rate. Secondly, a test cannot be taken as a necessarily true indication of the yielding behaviour. Very slight misalignments of the specimen in the machine can cause the non-appearance of a yield-drop. To overcome this, specimens must be aligned by pre-straining before they are aged, and must be held under some load to maintain their alignment during aging. A subsequent re-test can then be taken as a true indication of the effect that aging produces on the yielding behaviour. Thirdly, if the above conditions have been fulfilled and a yield drop has been produced, a further immediate re-test must be run to show that it is the aging treatment alone which has caused the yield drop. If the latter is the case no yield drop should be found in the "over-straining" test. All of these procedures were used in the present experiments.

In the first series of tests, crystals grown from arc-cast stock were used. It was considered that these crystals contained sufficient carbon to produce a yield point and that they would

therefore provide some indication of the magnitude of the strain-aging effects to be expected in later experiments with the purer material. Results typical of numerous samples examined are shown in Fig. 4. The initial alignment pre-strain at room temperature was followed by aging at 650°C for 1 hr., but this treatment failed to produce a yield drop or discontinuity in the stress-strain curve. The curve obtained after aging was in fact exactly similar in character to that obtained on re-straining without aging. Two further aging treatments produced the same negative result.

The inability of the "as-grown" carburized crystals to strain-age under the conditions described above was consistently observed, and came as a surprise. Two explanations for the result seemed possible, either that the aging treatment had allowed insufficient diffusion of carbon to the dislocations, or else that very little of the carbon in the crystals was contained in solid solution. Calculations based on published diffusion data (10) indicated, by elimination, that the latter explanation was the more likely one. No exact figures are available for the room temperature solid solubility of carbon in molybdenum but an approximate solid solubility limit of 50 ppm carbon has been reported (11) for a temperature of 1600°C. This suggests that the room temperature solid solubility must be extremely low. The conditions of crystal growth would favour carbide precipitation since the crystal behind the molten zone cools very slowly to room temperature.

To test the above hypotheses, a number of "as-grown" carburized crystals were solution-treated by annealing them at high temperature and then fast cooling. The anneals were made in the electron-beam apparatus at a temperature of 2000°C (the data of Spacil and Wulff (12) indicate that carbide dissolution takes place quite rapidly at this temperature) by supporting each specimen vertically in the centre of the electron gun, on a ceramic pedestal of low thermal conductivity. In this way reasonably uniform specimen temperatures were maintained for 15 mins., after which time the power was switched off. Cooling to below red-heat took about 0.5 mins. To avoid surface contamination effects, the anneals were made before samples were electro-machined and polished.

A set of results for a typical solution-treated crystal is given in Fig. 5. Smooth yielding was observed in the initial alignment test, but a sharp yield drop (about 12-1/2% reduction in stress) appeared after aging for 1 hr. at 650°C. This yield drop was absent in an immediate re-test but reappeared on further aging. An immediate re-test again resulted in a smooth curve. The tests illustrate a behaviour which is characteristic of a material that shows true strain-aging effects.

After establishing the conditions for strain-aging in the

crystals prepared from arc-cast stock, attention was directed to experiments with the purer crystals prepared from sintered material. The object of these experiments was firstly to provide a further demonstration that carbon was the source of the strain-aging in less pure crystals, and secondly to determine whether the purer crystals would be suitable for use as a base material in later experimental studies of the strain-aging effects produced by adding oxygen and nitrogen to molybdenum. Tests were made on both "as-grown" and solution-treated materials.

Results for a typical "as-grown" crystal are shown in Fig. 6. Two separate aging treatments (1 hr. at 650°C.) failed to produce a yield drop or any sign of strain-aging in this crystal, the only apparent effects of these treatments being to slightly reduce the flow stress. Fig. 7 shows similar tests for a crystal in the solution-treated condition. Here, an anneal of 1 hr. at 750°C (similar results were obtained when the annealing was done at 650°C) following the alignment pre-strain, produced no yield drop in the next test, but the flow stress was raised in this test and the form of the stress-strain curve indicated that some strain-aging had taken place. Smooth yielding occurred in the immediate re-test, and also in the following test which was made after aging the specimen for another 1 hr. at 750°C. A further annealing treatment of 3 hrs. at 750°C also failed to produce any discontinuity in the stress-strain curve. These experiments indicate two main points. Firstly, that carbon, the only impurity contained in appreciably greater amounts in the crystals grown from arc-cast material compared with those grown from sintered material, is the main source of the strain-aging exhibited by the former. Secondly, that the very slight strain-aging effects exhibited by the purer crystals can only be observed at low levels of strain. Reasons for the dependence of aging behaviour upon strain are discussed later.

In an effort to obtain further information concerning the interaction of carbon atoms and dislocations in molybdenum, a considerable time was spent in attempting to determine the kinetics of the strain-aging process in solution treated carburized crystals from studies of the yield drop return as a function of time and temperature. Initial experiments indicated that fully developed yield drops could be obtained in reasonable times (up to a few hours) at temperatures between 550°C and 750°C., but as soon as detailed studies were started it was found that the results obtained from different crystals were not very reproducible. The only apparent reason for the lack of reproducibility was a variation in the carbon content of the crystals, and no easy means was available for controlling this variable since its origin lay in

the starting material. However, it did appear likely that the problem could be by-passed if aging curves for several temperatures could be obtained by using just one specimen. Unfortunately, this approach also failed because the number of useful tests (about 10) that could be made on a single specimen was limited by the early onset of necking (at about 10-15% elongation). Currently, consideration is being given to other methods of determining aging kinetics. Resistivity and dynamic modulus techniques appear the most promising.

B. Effect of Pre-Straining and High Temperature Annealing on the Yield Strength of Molybdenum Crystals.

In previous tests (7) with molybdenum crystals grown from Wah-Chang stock, specimens pre-strained 5% and then annealed for 2-1/2 hrs. at 1500°C were found to possess a greater resistance to creep at 1000°C than duplicate specimens which were annealed without pre-straining. Micro-structural examinations indicated that a sub-structure was the source of the strengthening in the pre-strained and annealed material. As a logical extension of these experiments the present work was aimed at an evaluation of the effect of pre-straining and annealing on the mechanical properties of molybdenum when carbon is present as an impurity. Comparative tests were made on crystals grown from arc-cast and sintered stock, using sets of two or three samples prepared from the same "as-grown" rod. One sample from each set was pre-strained in tension at room temperature to the maximum allowed (about 10-15%) by the onset of necking, the second to approximately one half of the maximum value, and the third, when available, was left un-strained. All of the samples in a set were then annealed together for 2 hrs. at 1500°C and slow-cooled to room temperature. Further room temperature tensile tests were used to evaluate the mechanical property changes produced by the various treatments.

Fig. 8 shows typical results for a set of carburized crystals. The first "as-grown" specimens yielded smoothly at a resolved shear stress of 11,000 psi and was strained 13%. After annealing, a small yield drop appeared and the yield strength (lower yield value) was raised to 20,000 psi. The yield strength of the second "as-grown" specimen was 10,300 psi and it was strained 7%. It also showed a small yield drop after annealing and an increase in yield strength to 16,100 psi. The third specimen, tested after annealing without pre-strain, yielded at a stress of 10,500 psi, a value close to those for the "as-grown", unannealed samples. Analysis of one of the crystals from this series gave the carbon content of 76 ppm.

Results typical of the purer crystals grown from Wah-Chang

stock are given in Fig. 9, which shows tests on two crystals, both of which were pre-strained. Following an 8% pre-strain and the standard annealing treatment, the yield strength of the first crystal was raised from 9,400 to 11,500 psi. The same annealing treatment on the second crystal, which was pre-strained 4%, raised the yield strength from 12,000 to 13,000 psi. The analysed carbon content of one of the crystals of this series was 4 ppm.

Table II lists for the tests of Figs. 8 and 9, the relative percentage changes in yield strength produced by the various pre-straining and annealing treatments, and in Fig. 10 these relative yield strength changes are plotted as a function of percentage pre-strain for the two materials. A nearly linear dependence of the increase in yield strength upon percentage pre-strain is observed in each case, but for a given pre-strain the yield strength of the material of high carbon content is raised almost three times as much as that of the purer material.

Metallographic examinations of the specimens were made and, apart from the observation that none of the specimens recrystallized, the results must be considered as tentative. It appears that all of the pre-strained and annealed crystals contain a sub-structure, the density of which increases with increasing pre-strain. The sub-grain size in the 13% pre-strained carburized crystal is about 10 μ.

IV. DISCUSSION:

A. Strain-Aging Experiments.

It is normally assumed, and generally quite validly so, that the mere introduction of a suitable quantity of an interstitial impurity into an otherwise pure body-centred-cubic metal is sufficient to produce strain-aging effects. The most important finding of the present strain-aging experiments is that for the case of carbon in molybdenum this assumption is incorrect. Quantities of carbon more than sufficient to saturate normal densities of dislocations in molybdenum are apparently, under equilibrium conditions at moderate temperatures, contained in the material almost solely as precipitated carbides, and as such are not available for strain-aging. To make them available the material must first be subjected to a rather difficult high temperature annealing treatment and then fast cooled.

The strain-aging behaviour of solution-treated carburized molybdenum follows the same general pattern as that of other impure body-centred-cubic metals. Relatively high temperatures (550°C - 750°C) are, however, required to produce aging effects in reasonable times and, in magnitude, these effects (as shown, for example, by the size of the fully developed yield drop) are

somewhat smaller than those observed in iron, tantalum and niobium. This may be because carbon atoms arriving at dislocations in molybdenum, re-precipitate as carbides early in the aging process, relieving the local stresses and thus removing the driving force for further aging. Early carbide precipitation on dislocations is certainly to be expected in view of the low solubility of carbon in molybdenum.

As yet, no study has been made in the current programme of the strain-aging produced in molybdenum by oxygen and nitrogen. It seems likely, however, that these impurities will behave in much the same way as carbon since they both exhibit a low solid solubility in molybdenum at moderate temperatures (11) and both form compounds which can only be dissolved at relatively high temperatures (12). Also, the results of strain-aging studies (13) (14)(15) on various grades of commercial molybdenum containing oxygen and nitrogen (as well as carbon) indicate relatively slight strain-aging effects. In the latest of these studies (15) the low solubility of the interstitial impurities was suggested as the reason for this behaviour, although no tests were made on solution-treated materials to confirm the suggestion. It is of general interest to note here that tungsten is also generally reported as showing rather small strain-aging effects. The reason again, as with molybdenum, is probably not that interstitial impurities do not interact strongly with dislocations in the tungsten lattice, but it is more likely that there are generally only few dissolved interstitials available for interaction.

With the molybdenum crystals grown from Wah-Chang stock only small strain-aging effects were observed, even after a solution treatment, and these effects disappeared during testing as the strain was increased. A simple calculation reveals that these results are compatible with the analytical determinations of a 20 ppm average interstitial impurity content for the crystals. Thus, even if all of the impurities were available for strain-aging they would still, assuming $10^{10}/cm^2$ as a reasonable dislocation density after initial pre-strain of a few percent, be able to supply little more than one interstitial atom to each atom plane of dislocation during the first aging treatment. After this treatment negligible amounts of impurity would remain for aging in subsequent tests.

One further general conclusion from the experiments should be emphasized. The results indicate quite clearly that the enhancement or suppression of strain-aging effects in molybdenum of a given purity can be best achieved by the choice of suitable initial annealing procedures. This factor should be taken into account in the design of practical fabrication procedures.

B. Pre-straining and Annealing Experiments.

In previous studies (7) it was shown that pre-straining and high temperature annealing treatments which produce a sub-structure in molybdenum, also result in a strengthening of the material. The present experiments have confirmed these findings and have further shown that the degree of strengthening increases with increasing pre-strain and increasing impurity (carbon) content. These new results strongly suggest that the sub-grain boundaries in molybdenum act in the same qualititive way as ordinary grain boundaries, at least regarding their general influence on room temperature yield strength.

Numerous experiments have shown that the yield strength σ_y of iron and the body-centred-cubic transition metals varies with grain size 2d, according to a relationship of the type

$$\sigma_y = K + K_1 \cdot d^{-1/2}$$

This relationship has been justified on theoretical grounds by Petch (16) and Cottrell (17) who envisage yield propagation as a process in which deformation is transferred from grain to grain under the action of stress concentrations produced by dislocations piled-up at grain boundaries. Thus, consider a material in which a few grains have yielded to produce pile-ups at either boundary. For a grain size 2d the average pile-up length will be d and at an applied stress σ_y the effective stress acting on the pile-ups will be $(\sigma_y - \sigma_i)$ where σ_i is the (friction) stress resisting the movement of dislocations across their slip planes. Dislocation theory gives the stress at distance l ($l \ll d$) ahead of the pile-ups as $(\sigma_y - \sigma_i)(\frac{d}{l})^{1/2}$, and for yield to propagate this stress must equal the value σ_D required to operate the nearest sources (those at distance l) in adjacent grains. Equating, we obtain

$$(\sigma_y - \sigma_i)(\frac{d}{l})^{1/2} = \sigma_D$$

or, alternatively

$$\sigma_y = \sigma_i + \sigma_D \, l^{1/2} \cdot d^{-1/2} \qquad (1)$$

Now, if sub-grain boundaries in molybdenum act in the same way as grain boundaries by causing dislocations to pile-up during deformation, Equation 1 should describe the yielding behaviour of sub-structured material, and should provide a consistent explanation for the present results. We find in fact that it does this. Thus, the strengthening of "as-grown" crystals can be explained as the result of introducing barriers where none (or few) existed previously; the increase in strengthening with increasing pre-strain as the result of decreasing the value of d by decreasing the sub-grain size; the increased strengthening with increased impurity content as the result of a raising of σ_p by carbon locking.

It should be noted that the last of the explanations given above is based on two assumptions; that annealing at 1500°C allowed precipitated carbon to pass into solution and then to segregate to dislocations, and that this redistribution of the carbon did not produce appreciable changes in σ_i .For the present experiments, these assumptions can be justified; the first by the observation that yield drops appeared in the tests on the carburized crystals after annealing, and the second by the observation, throughout all of the experiments, that carbon does not produce appreciable lattice friction strengthening in molybdenum, regardless of whether it is present in solid solution or as gross carbides. For molybdenum containing other interstitial impurities, however, similar arguments may not apply. Thus, an extreme situation can be imagined in which pre-straining and annealing produces more weakening by lowering σ_i (through a change in impurity distribution), than it does strengthening by introducing sub-boundaries. It is believed, in fact, that such a situation has already been observed in experiments on crystals grown from G.E. sintered stock. The crystals were initially quite strong (stronger than those grown from Wah-Chang or Climax arc-cast stock) but after pre-straining and annealing the yield strength was lowered by an amount which increased with increasing pre-strain. Currently the crystals are being analysed to see if the results can be correlated with any specific interstitial impurity.

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FIG. I. ELECTRON GUN ASSEMBLY FOR (a) NORMAL RUNNING AND (b) RUNNING SPECIMENS OF HIGH GAS CONTENT.

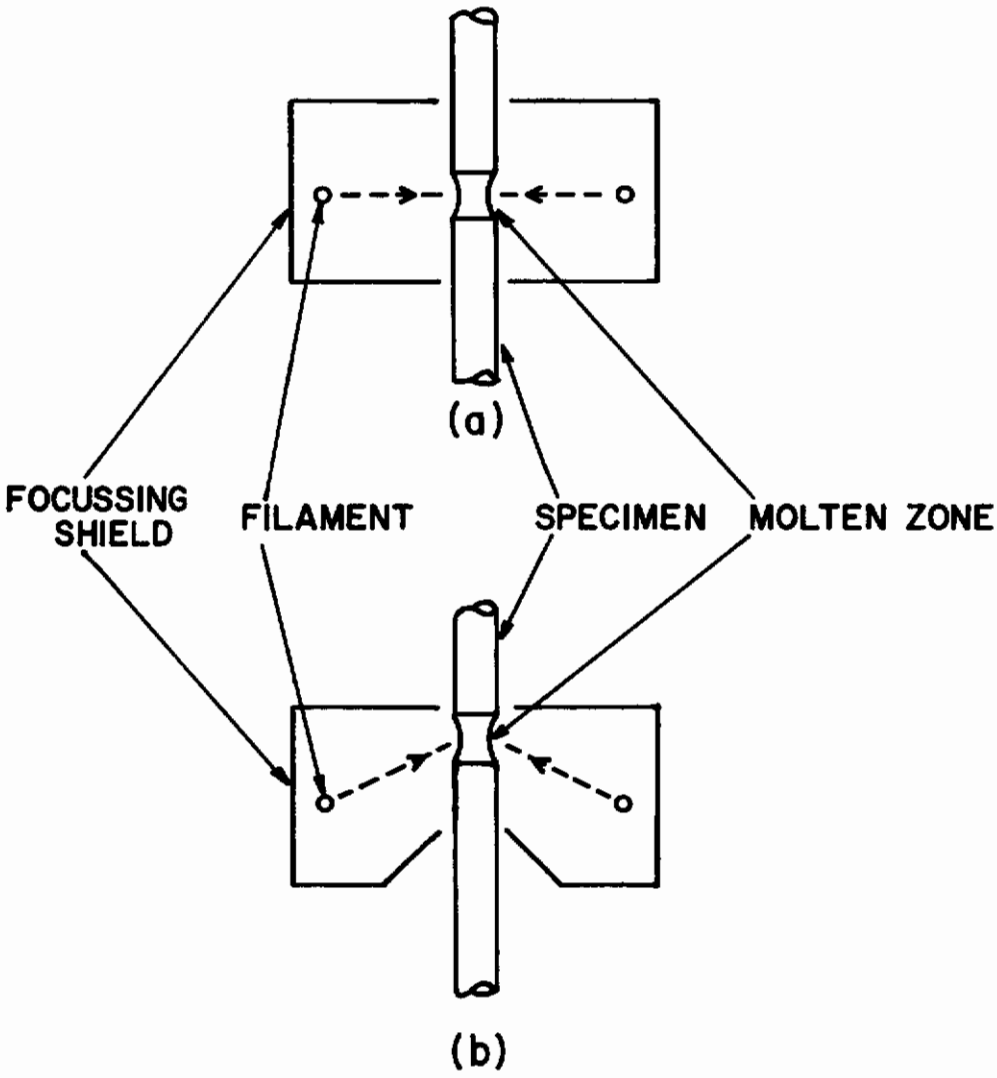


FIG. 2. ORIENTATIONS OF THE CRYSTALS USED IN THE TESTS

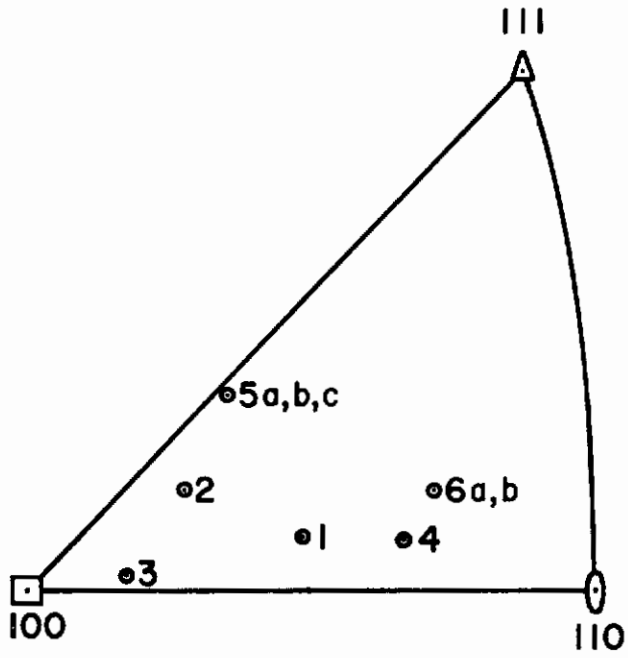


FIG. 3. HIGH TEMPERATURE STRAIN-AGING ASSEMBLY

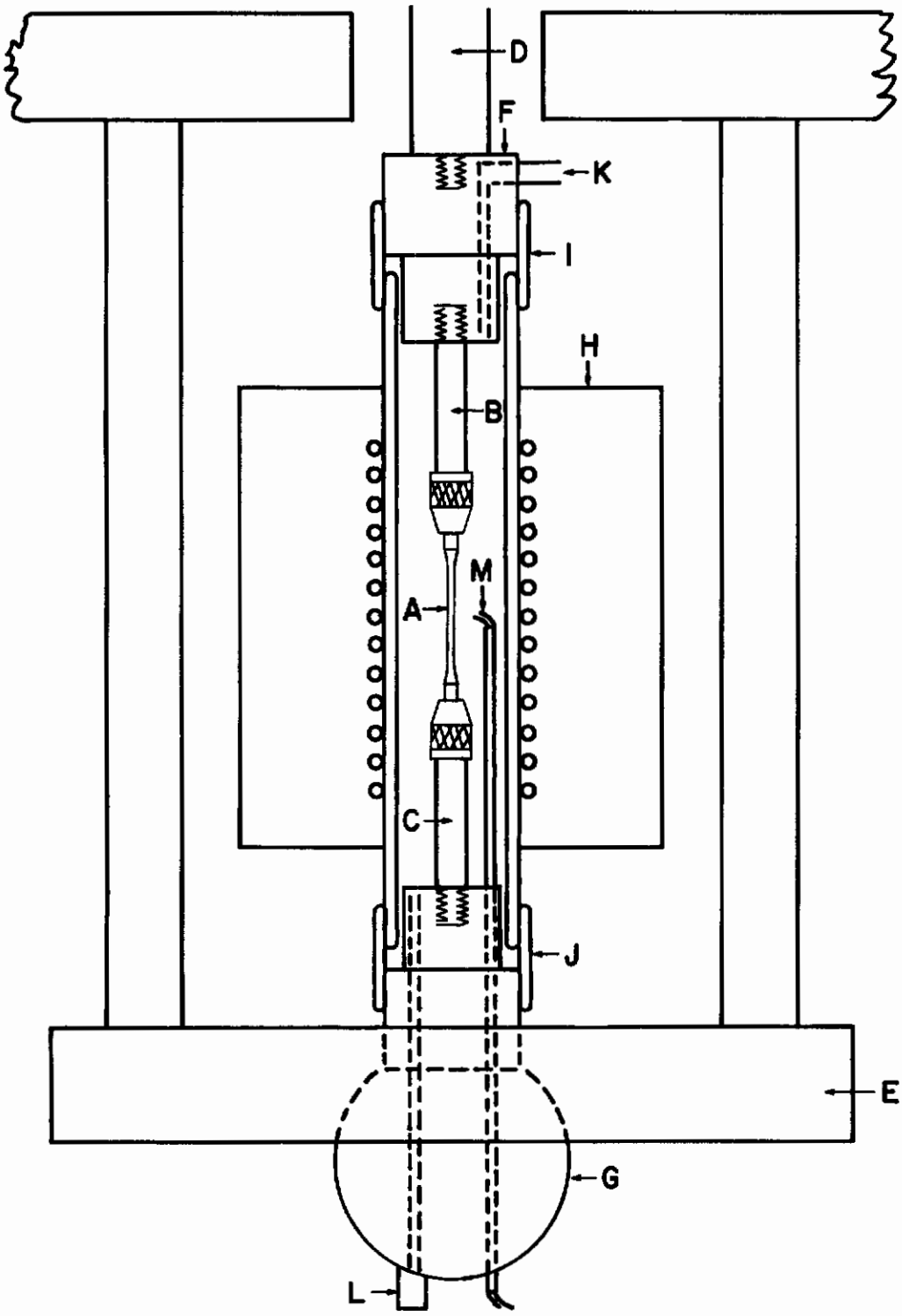
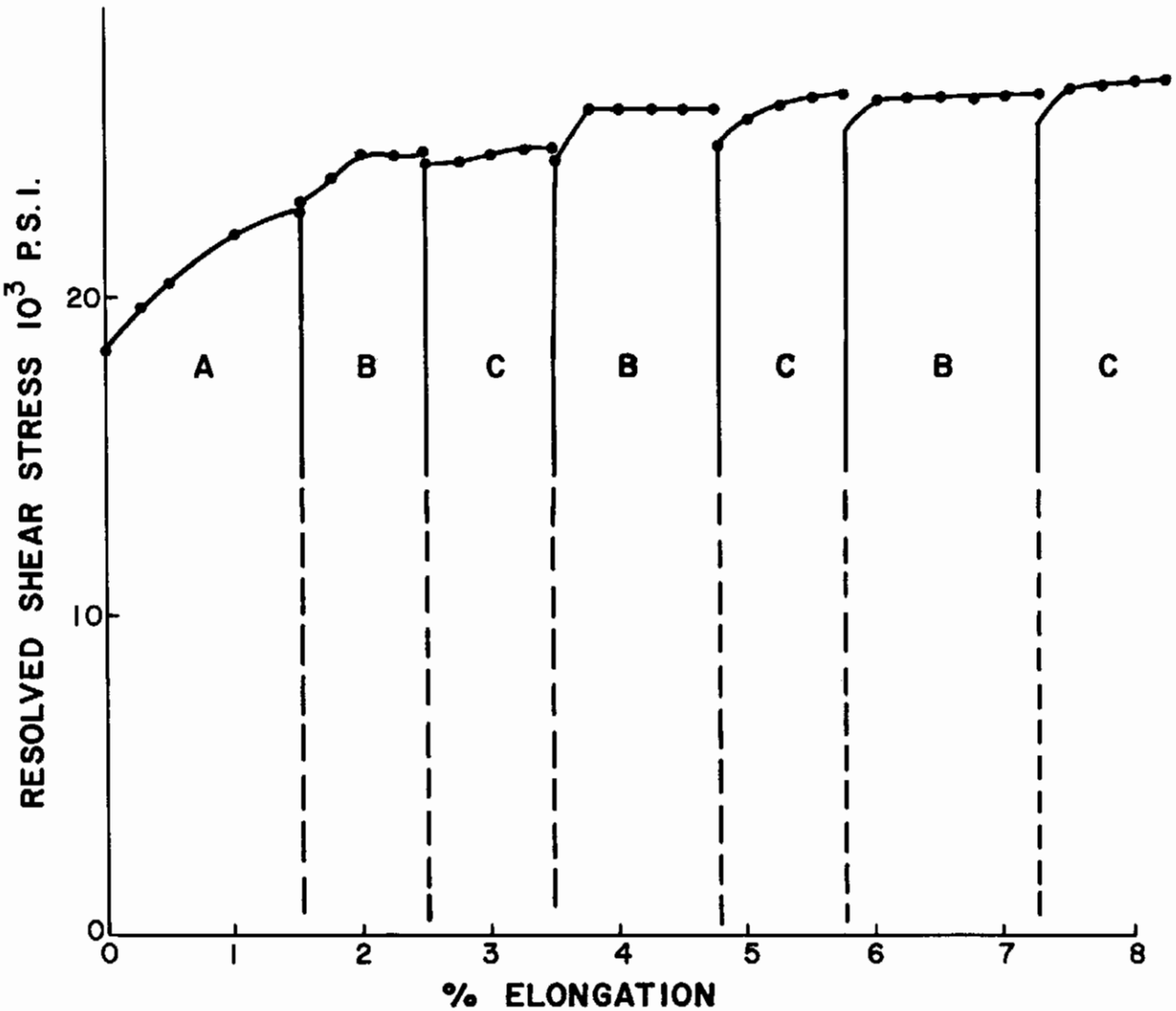


FIG. 4. STRESS-STRAIN CURVES FOR A MOLYDENUM CRYSTAL (SPEC. NO. I) GROWN FROM CLIMAX ARC-CAST STOCK.



A. "AS-GROWN"

B. AGED I HR. 650° C

C. IMMEDIATELY AFTER B

ALL TESTS AT ROOM TEMPERATURE

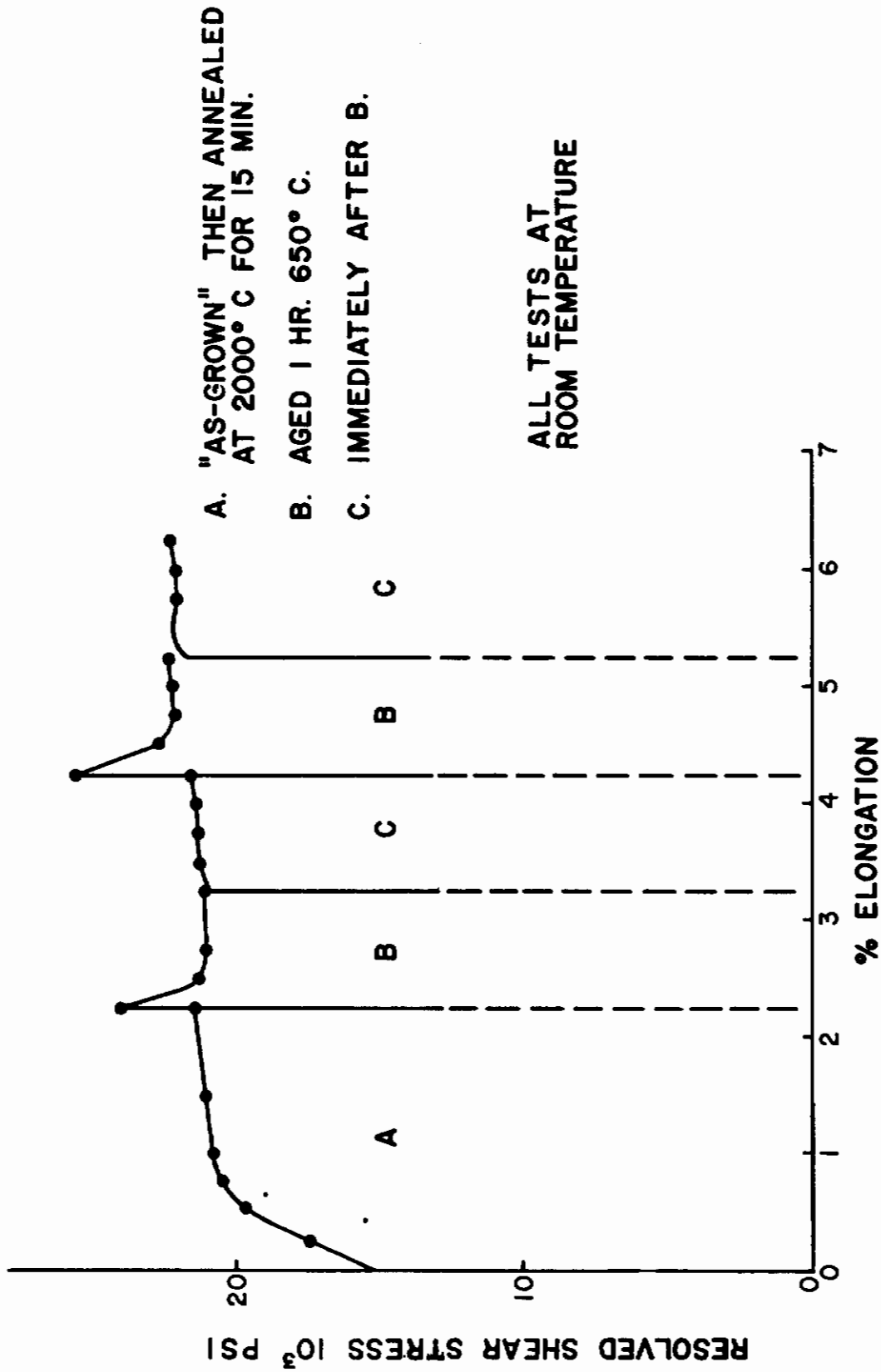


FIG. 5. STRESS-STRAIN CURVES FOR A MOLYBDENUM CRYSTAL (SPEC. NO. 2) GROWN FROM CLIMAX ARC-CAST STOCK AND SOLUTION TREATED.

FIG. 6. STRESS-STRAIN CURVES FOR A MOLYBDENUM CRYSTAL (SPEC. NO. 3) GROWN (DOUBLE PASS) FROM WAH CHANG SINTERED STOCK.

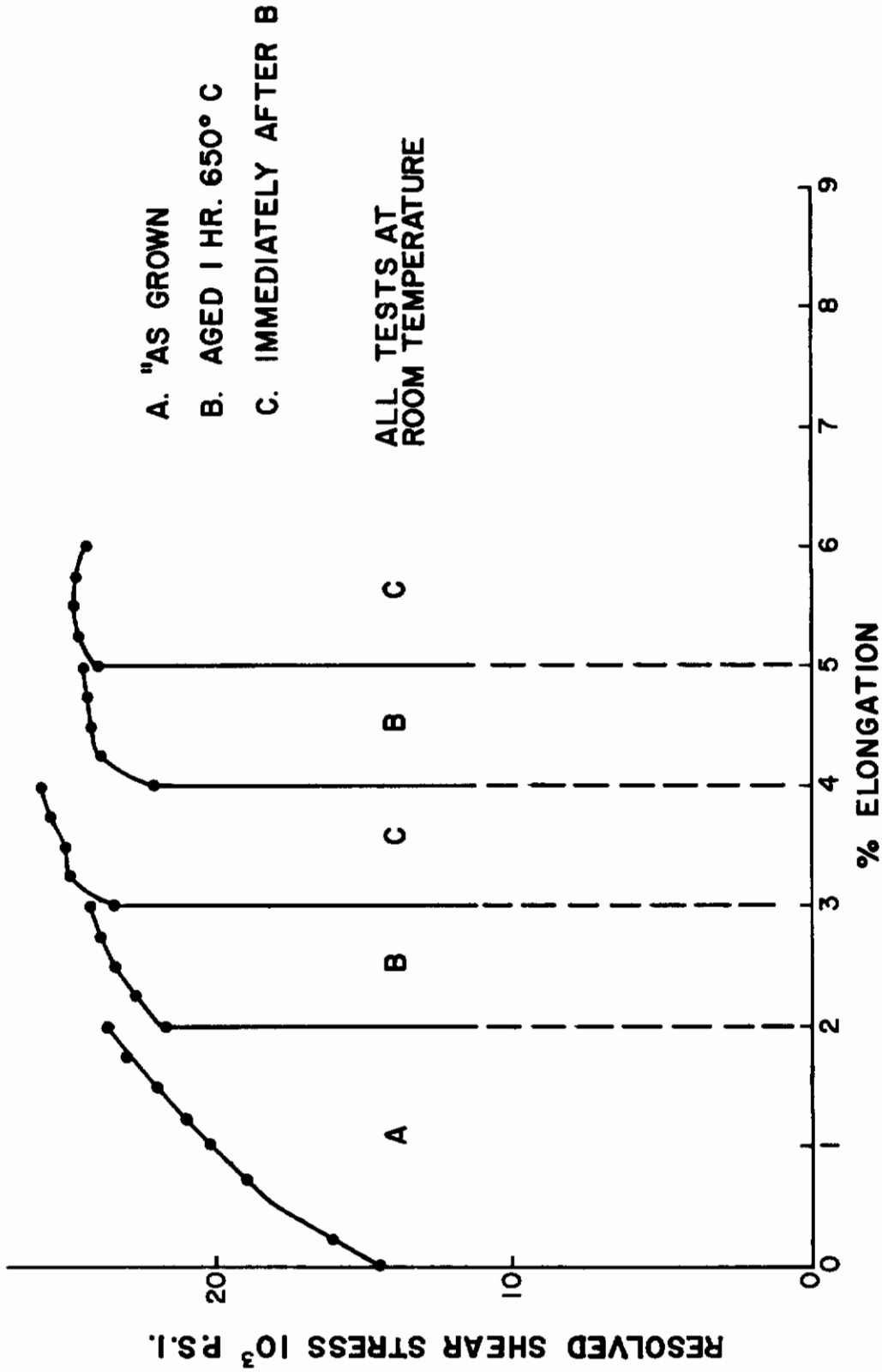


FIG. 7. STRESS-STRAIN CURVES FOR A MOLYBDENUM CRYSTAL (SPEC. NO. 4) GROWN (DOUBLE PASS) FROM WAH CHANG SINTERED STOCK AND SOLUTION TREATED.

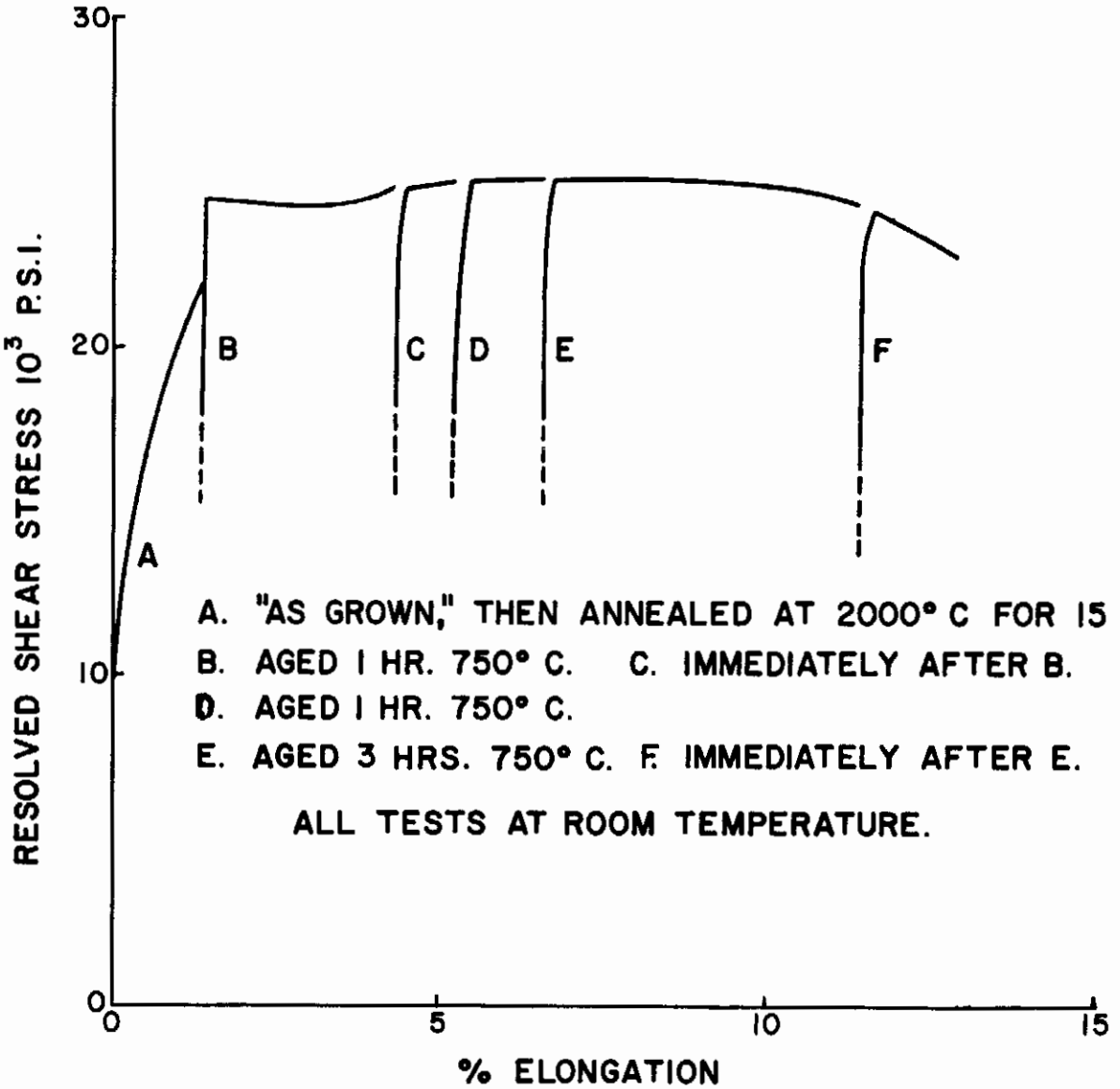


FIG. 8. EFFECT OF PRE-STRAINING & HIGH TEMPERATURE ANNEALING ON THE YIELD STRENGTH OF MOLYBDENUM CRYSTALS GROWN FROM CLIMAX ARC-CAST STOCK.

A_1. SPEC. 5a, "AS GROWN" A_2. ANNEALED 2 HRS. AT 1500° C AFTER A_1.
B_1. SPEC. 5b, "AS GROWN" B_2. ANNEALED 2 HRS. AT 1500° C AFTER B_1.
C_1. SPEC. 5c, "AS GROWN", THEN ANNEALED 2 HRS. AT 1500° C.

ALL TESTS AT ROOM TEMPERATURE.

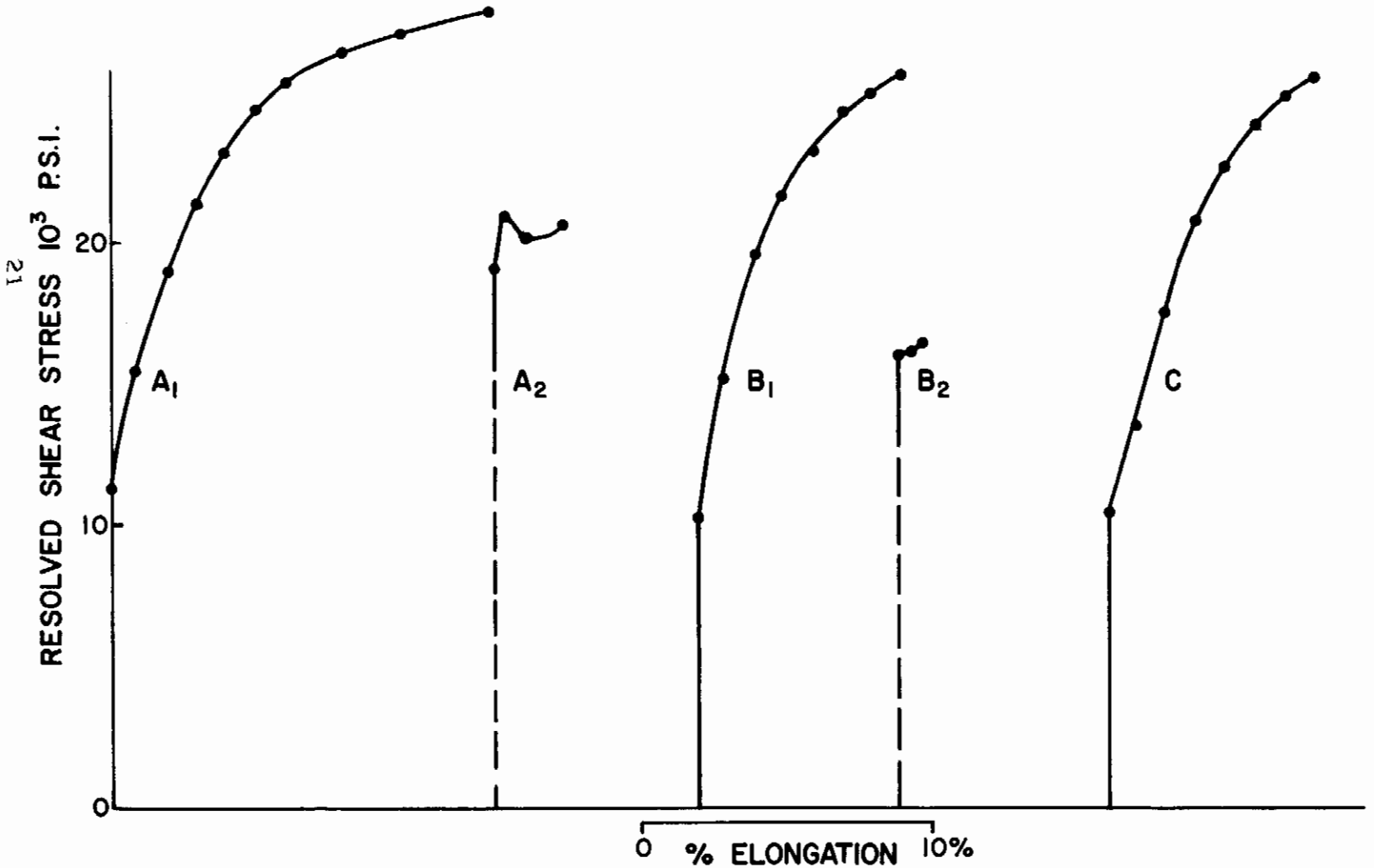
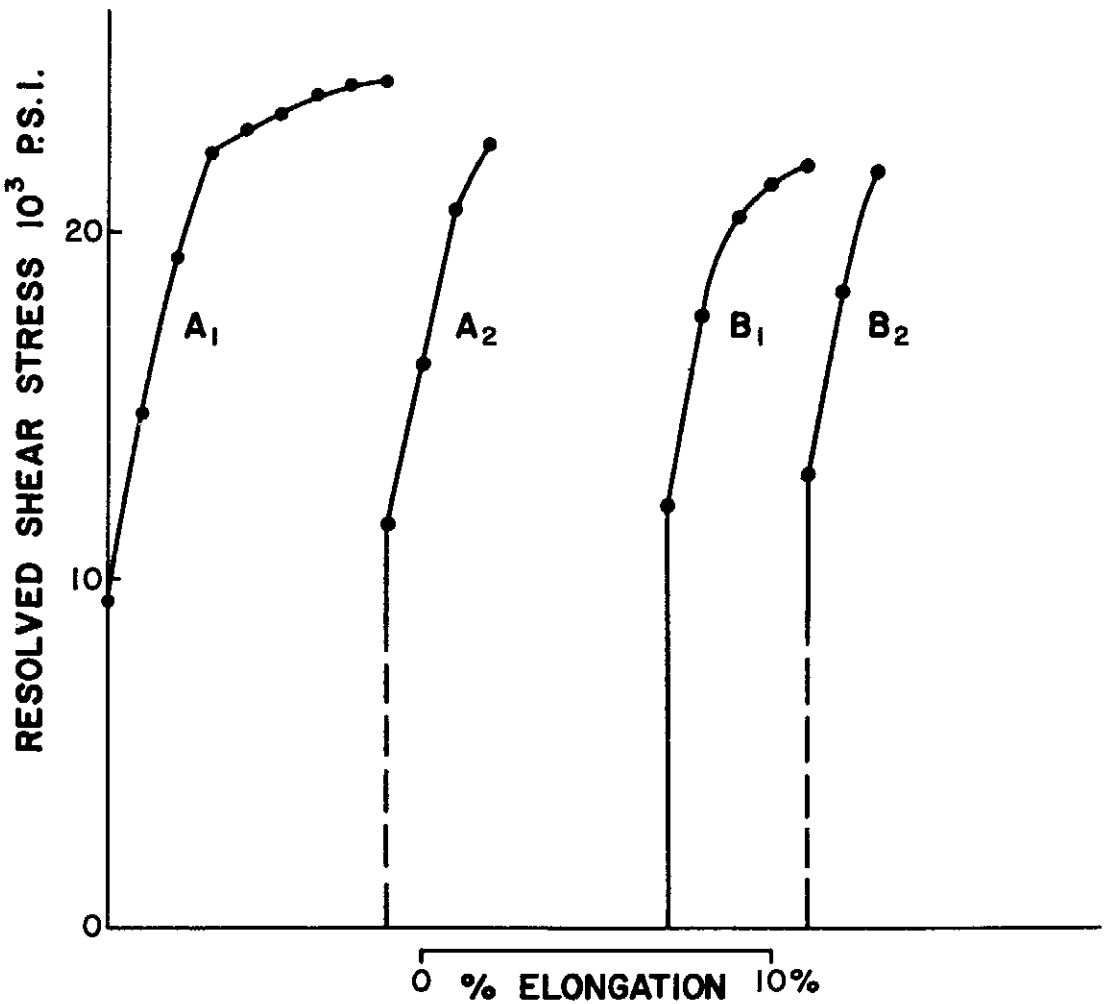


FIG. 9. EFFECT OF PRE-STRAINING & HIGH TEMPERATURE ANNEALING ON THE YIELD-STRENGTH OF MOLYBDENUM CRYSTALS GROWN (DOUBLE PASS) FROM WAH-CHANG SINTERED STOCK.

A_1. SPEC. 6a, "AS GROWN." A_2. ANNEALED 2 HRS. AT 1500° C AFTER A_1.

B_1. SPEC. 6b, "AS GROWN." B_2. ANNEALED 2 HRS. AT 1500° C AFTER B_1.

ALL TESTS AT ROOM TEMPERATURE



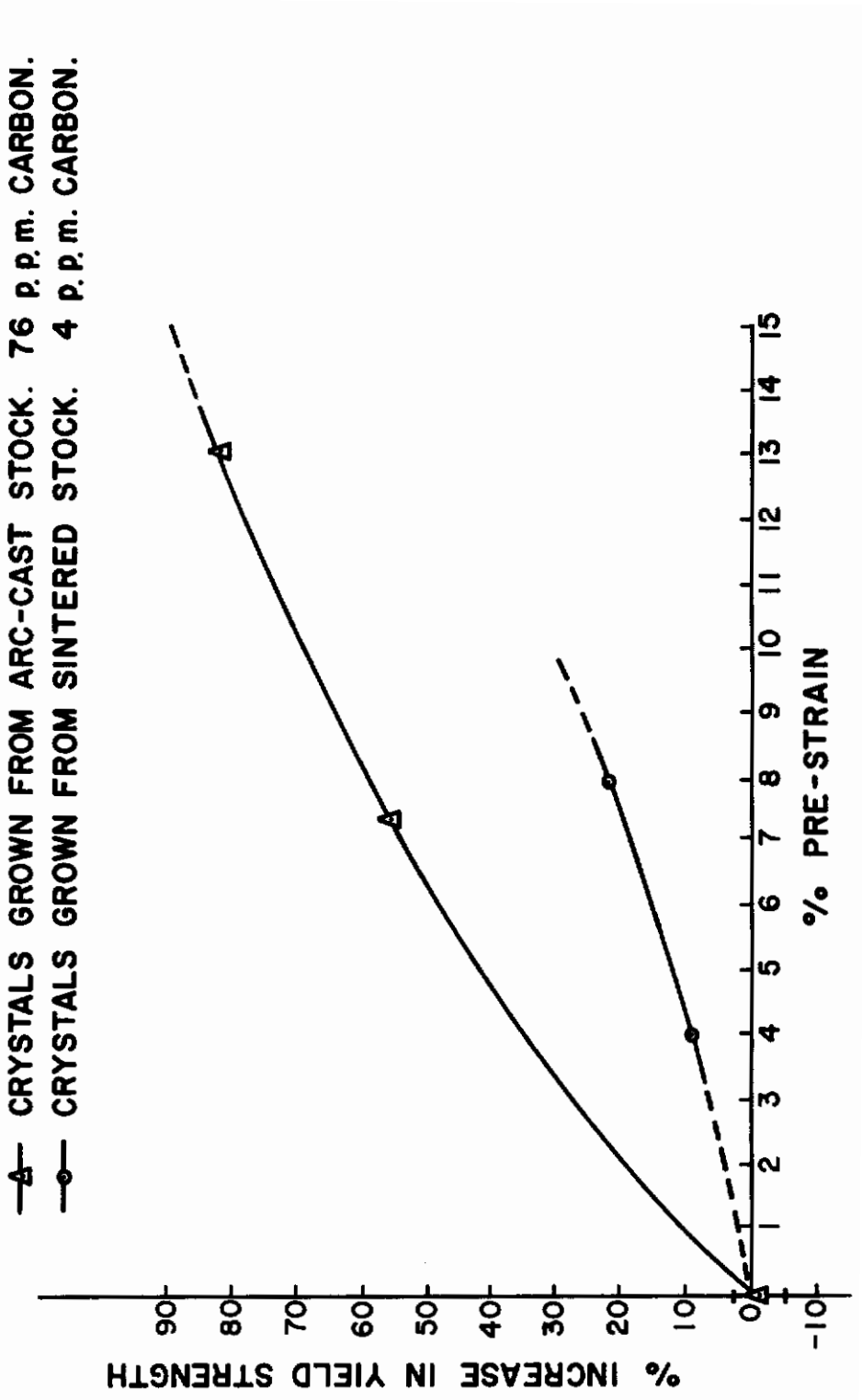


FIG. 10. EFFECT OF CARBON CONTENT ON THE INCREASE IN YIELD STRENGTH PRODUCED BY PRE-STRAINING & ANNEALING (2 HRS. AT 1500° C) MOLYBDENUM CRYSTALS.

TABLE I

Interstitial Impurity Contents of Typical Molybdenum Specimens

MATERIAL	IMPURITIES wt.ppm			
	O_2	N_2	H_2	C
Wah-Chang Sintered Stock (as received)	100-150	25-50	2-5	50-100
Beam Melted Wah-Chang Stock (double pass at 3"/hr.)	5-10	2-5	0-2	4-15
Climax Arc-Cast Stock (as received)	15	20	5	100-400
Beam Melted Climax Arc-Cast Stock (single pass at 3"/hr.)	5-11	0.5-5	0-2	75-350

TABLE II

Relative Percentage Changes in Yield Strength Produced by Pre-Straining and Annealing (2 hrs. at 1500°C) Molybdenum Crystals of Different Carbon Content.

Material	Beam Melted Climax Arc-Cast Stock 75 ppm			Double-Pass, Beam Melted Wah-Chang Sintered Stock 4 ppm Carbon		
% Pre-strain	0	7	13	0	4	8
% Change in Yield Strength	+1.94 to -4.55	+56.31	+83.64	-	+8.33	+22.34