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ALLOY SINGLE CRYSTALS**

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L. I. Van Torne and G. Thomas

June 1965

STRUCTURE AND MECHANICAL PROPERTIES OF Ta-Mo ALLOY SINGLE CRYSTALS

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ABSTRACT

High purity single crystals of the Ta-Mo alloy series were grown near 10 atomic percent increments by electron beam zone refining. The methods of investigation of the prepared crystals included: specific resistivity at 273°K, 77°K and 4.2°K, critical resolved shear stress for plastic flow or cleavage at 273°K, lattice parameter determinations, transmission electron microscopy and quantitative X-ray diffracted intensity measurements of {200} and {220} reflections.

It was found that alloys of compositions between Mo-19 Ta and Mo-58 Ta cleaved on the {100} planes. The lattice parameter measurements and transmission electron microscopy showed that the alloy series is bcc with no long range order coherent over dimensions greater than 300Å, nor intermediate phases. Kikuchi electron diffraction proved that there exist deviations from random solid solution in the brittle crystals. This result was confirmed by X-ray intensity measurements of 200 and 220 lines. The ratio of constituent atoms on {100} and {110} differ from the ratio of atoms corresponding to the total composition of the crystal in such a way as to generate quasi-cubic domains for {100} and quasi-dodecahedral domains for {110} deviations. It is suggested that the increase in critical resolved shear stress for plastic flow with alloy composition results from a corresponding increase in the Peierls-Nabarro friction of dislocations produced by alloying, composition deviations

[†]Now with the Martin Co., Orlando, Florida.

V		VI	
AT.NO. 40	AT.NO. 41	AT.NO. 41	AT.NO. 74
Rad.1.43 Wt. 929	Rad.1.36 Wt. 95.9	Rad.1.36 Wt. 95.9	Rad.1.37 Wt. 183.9
Nb		Mo	
BCC M.P. 2415		BCC M.P. 2622	
$4p^6 4d^4 5s$		$4p^6 4d^5 5s$	
AT. NO. 73	AT. NO. 74	AT. NO. 74	AT. NO. 74
Rad.1.43 Wt.180.9	Rad.1.37 Wt.183.9	Rad.1.37 Wt.183.9	Rad.1.37 Wt.183.9
Ta		W	
BCC M.P. 2996		BCC M.P. 3395	
$5d^3 6s^2$		$5d^4 6s^2$

MECHANICAL BEHAVIOR NEAR 1-1 COMPOSITIONS

MUB-4438

Table I

Of the four binary bcc solid solution alloys of interest which show the cleavage behavior, viz., Nb-W, Nb-Mo, Ta-W and Ta-Mo, the binary alloy system Ta-Mo was selected for detailed investigation. This alloy system was chosen because of the large differences in the respective X-ray and electron scattering factors and the closeness of the melting points of the two constituents. The latter factor facilitates single crystal preparation by electron beam zone refining.

2. EXPERIMENTAL AND RESULTS

Several series of experiments were carried out and for brevity the details of the experimental procedures and apparatus for zone refining, crystal preparation, resistivity and X-ray intensity measurements will not be given here, but are available.⁽⁴⁾

2.1 Preparation of the Alloy Single Crystals

In investigations of body-centered-cubic metallic crystals special consideration must be given to the preparation of the alloys so as to minimize interstitial impurities. An electron beam zone refiner^(5,6) was designed and constructed⁽⁴⁾ in order to prepare ultra pure alloy single crystals.

Several different diameter rods of Ta and Mo of commercial purity were obtained and a combination of rod diameters was selected to achieve the desired alloy composition (see Table II). The rods were bound together with 0.5 mm Ta wire and zone refined in the following manner.

1. Three passes (2.54 cm/min) at a temperature 200°C below the melting point of the alloy to remove as much residual gas as possible prior to melting.

TABLE II

Alloy	Specific Resistivities ($\mu\Omega\text{cm}$)			Lattice Parameters ($\text{\AA}$)
	$\rho_{273^\circ\text{K}}$	$\rho_{77^\circ\text{K}}$	$\rho_{4.2^\circ\text{K}}$	a_0 (296°K)
Mo	5.02	0.435	0.00588	3.1460
Mo-7 Ta	7.43	3.22	2.61	3.1562
Mo-19 Ta	11.8	7.76	7.16	3.1783
Mo-28 Ta	17.3	12.8	11.9	3.2058
Mo-40 Ta	21.2	15.9	14.9	3.2242
Ta-50 Mo	25.7	19.4	18.0	3.2345
Ta-42 Mo	25.5	19.1	17.7	3.2469
Ta-30 Mo	23.0	15.7	13.7	3.2702
Ta-21 Mo	20.5	11.6	9.35	3.2837
Ta-12 Mo	19.9	10.1	7.54	3.2948
Ta	12.2	2.34	0.00127	3.3025

2. One fusion pass (2.54 cm/min) which eliminates much of the impurities.
3. Two zone passes along both directions of the rod.

The vacuum system employed was sufficient to maintain the pressure within the chamber at less than 10^{-5} torr during the first fusion pass. After the first pass the pressure remained near 10^{-6} torr.

The level of purity obtained by zone refining was demonstrated by a measured resistivity ratio ($\rho_{273^\circ\text{K}}/\rho_{4.2^\circ\text{K}}$) of 10^4 on a Ta crystal which corresponds to a residual level of interstitials equal to about 3 impurity atoms in 10^9 matrix atoms.^(4,7) The orientations were determined from back reflection Laue patterns and are shown in Fig. 3(a). The patterns showed that the crystals were monocrystalline and free from small angle boundaries.

To make specific resistivity and critical resolved shear stress measurements, it was necessary to have a uniform cross section over a considerable length of crystal. Since the as grown crystals were not uniform in section to the degree desired, crystals were cut to the desired length with a diamond saw, carefully mechanically polished on a rotary wheel using 240 grit emery cloth and finally electropolished in a $\text{H}_2\text{SO}_4\text{-H}_2\text{O}$ solution to remove the surface damage. The final diameter of the crystals were 2.30 mm (± 0.1 mm). This technique enables tensile specimens to be shaped without damaging the crystals.

2.2 Resistivity Measurements

Electrical resistivity measurements indicate the presence of second phases, intermediate compounds, and ordering through the appearance of inflection points or plateaus in a plot of resistivity versus composition.

The specific resistivities of the alloys under investigation were determined at 273°K, 77°K and 4.2°K, and the results are listed in Table II.

Resistivity measurements were made by the IR drop method employing a Rubicon 6 dial thermoc free potentiometer, Astrodata nanovoltmeter for null detector and a constant (± 1 mA drift/hr) current supply.

For resistivity measurements at 273°K, an ice and distilled water bath was used. A liquid nitrogen bath was used for the 77°K measurement and a Hofman ten liter liquid He dewar was used for the 4.2°K measurement. In all measurements the dewar probe with attached potential probe fixture remained in the temperature environment for a period of not less than twenty minutes prior to making the measurement. This procedure ensured that the crystal was at the same temperature as the enclosing media.

In order to measure the resistivity of the Ta crystal, it was necessary to increase the current to a value exceeding the superconducting critical current density. The superconducting transition temperature of Ta is 4.5°K and an increase from 5.510 to 8.920 amps, for this crystal cross section, was necessary to exceed the critical current density. When the current was increased to 9.582 amps, the crystal "decoupled" from the liquid helium as was indicated by an unsteady null.

Upon giving due consideration to the sensitivity of the electronic measurement instruments and the sensitivity of the dimension measuring instruments, the maximum error in each resistivity value shown in Table II is estimated to be less than one-half of one percent.

Figure 1 is a graph of the specific resistivities given in Table I versus alloy composition. It is immediately obvious from the figure that the specific resistivity at 273°K, i.e. $\rho_{273^\circ K}$, approximates to a

linear function of the atomic percent Ta up to and including 50 atomic percent Ta. The slope of this line is $0.394 \text{ } (\mu\Omega\text{cm})$. No such simple relationship is observed for Ta rich alloys.

The increase in resistivity arising from an increase in temperature from 4.2°K to 273°K is due mainly to an increase in the frequency and magnitude of electron wave scattering. Electron wave scattering events are proportional to the frequency and amplitude of atomic vibrations in the crystal. The amplitude of vibration of an atom in a crystal is directly related to the elastic constants of the crystal, i.e. the elastic compliances (and their inverse the elastic stiffnesses). Therefore, a graph of $\rho_{273^\circ\text{K}}$ minus $\rho_{4.2^\circ\text{K}}$ versus alloy composition is shown in Fig. 2. The observed abrupt changes in slope suggests that the compliances vary appreciably with composition and/or the density of electronic states is changing abruptly. In either or both cases, a simple linear variation is not obeyed.

2.3 Resolved Shear Stress Measurements

Tensile tests were made on all crystals at 273°K in order to determine the stress strain behavior.

The load versus elongation for those alloys which flow plastically together with the orientations of the tensile axes are shown in Fig. 3(b). The remaining crystals (Fig. 3(a)) all failed by cleavage. The load versus elongation is plotted rather than the true stress-true strain because the deformation along the gauge length was heterogeneous. Therefore, the tensile curves plotted in the figure were taken directly from the Instron tensile machine. It can be seen from Fig. 3(b) that the stress strain behavior of the crystals depends on orientation in a

similar way to fcc crystals. For example, the Mo crystal shows a three-stage stress strain curve, with an easy glide region beyond about 2% elongation. Similar behavior has been reported for single crystals of Nb⁽⁸⁾ and Fe.⁽⁹⁾

With the exception of Mo-7 Ta the crystals necked down rapidly and the cross section of the necked region was elliptical, indicating that at least two slip systems were operating after the initial plastic flow. However, only the Mo crystal shows a marked degree of work hardening. Alloys between 20 and 75% Ta were observed to cleave (Fig. 4) after a very small plastic elongation. The cleavage plane was determined to be {100} from Laue patterns taken after failure of the crystals. Assuming that the active slip systems in the alloys are {110}<111>, the critical resolved shear stress for slip was calculated. The maximum shear stress on the cleavage plane was also calculated and the data is shown in Table III and in Fig. 4. It can be seen that the solid solution strengthening is appreciable for the ductile alloys.

2.4 Lattice Parameters at 296°K

The binary alloy series tantalum-molybdenum is reported^(1,10) to form body centered cubic solid solutions at all compositions. In view of the mechanical property results a detailed examination of the X-ray spectra and lattice parameters as a function of alloy composition was made to determine the possible existence of second phases, ordering reactions, or anomalous changes in lattice parameter which might be related to the observed tensile behavior. Alloy powders were prepared by filing, but the filings were not subsequently annealed in order to avoid possible contamination. The previous investigators^(1,10) used

TABLE III Shear Stress for Flow or Cleavage

<u>Alloy</u>	<u>Slip on {011}<111>, Kg/mm²</u>	
Mo	3.63	
Mo-7 Ta	5.44	
Ta	5.10	
Ta-12 Mo	13.3	
Ta-21 Mo	14.2	
Ta-30 Mo	26.8	
	<u>Cleavage on {100}, Kg/mm²: τ on {011}<111></u>	
Mo-19 Ta	11.0	7.70
Mo-28 Ta	5.06	27.1
Mo-40 Ta	7.76	6.60
Ta-50 Mo	8.90	2.60
Ta-42 Mo	11.4	3.58

sintered powders which probably contained a relatively high level of residual impurities.

An X-ray diffractometer, rather than a film camera, was employed to measure the lattice parameter because the alloy powders were in a cold worked condition and, thus, gave rise to broad diffraction lines which can be measured more accurately with a diffractometer than on films obtained from the traditional powder cameras.

The position of the X-ray diffraction line was determined as shown in Fig. 5. It is obvious from the figure that copper $K_{\alpha 1}$ and $K_{\alpha 2}$ are unresolved; therefore, the wavelength used in computation of the lattice parameters was the weighted average of these wavelengths, the weighted average being taken in the accepted manner, e.g. $\bar{\lambda} = \frac{2\lambda(\alpha_1) + \lambda(\alpha_2)(11)}{3}$.

The X-ray spectra of every alloy indexed as body centered cubic as previously reported. Extra or superstructure lines were searched for and unobserved over the total angular range of reflections. This verifies the observations of the previous investigators.

The lattice parameter computed from each spectral line measured for a given alloy was plotted against the Nelson-Riley function $\frac{1}{2} \left(\frac{\cos^2 \theta}{\sin \theta} + \frac{\cos^2 \theta}{\theta} \right)$. A straight line was drawn through the points and extrapolated to NRF = 0, the value of the intercept being the true lattice parameter.

The lattice parameters measured in this investigation are given in Table II and plotted as circles in Fig. 6. The lattice parameters reported by Geach and Summers-Smith⁽¹⁰⁾ are also shown and plotted as squares. The dotted line is a plot of Vegard's law for the data of

the latter investigators, and the solid line is for the data of this investigation. The previous work shows a negative deviation from Vegard's law which suggests the existence of some degree of short range order in this alloy series. However, our work shows a positive deviation which suggests some degree of clustering, i.e. a tendency for formation of a miscibility gap. A possible explanation of the negative deviation reported in the previous work is the presence of several atomic percent of dissolved interstitial impurities resulting from the use of sintered powders. Since the bond energy of the carbides of Ta and Mo are large, then the "bond length" between matrix atom and interstitial impurity, e.g. carbon, is expected to be short. Therefore, an interstitial impurity would tend to pull the surrounding matrix atom towards it and thereby produce an incremental decrease in the lattice parameter. Thus, the levels of interstitial impurities found in sintered powders of refractory metals could easily lead to the observation of a negative deviation in Vegard's law.

2.5 Transmission Electron Microscopy

Thin foils were prepared from the crystals used for the resistivity and tensile measurements using a special micropolishing technique described elsewhere.⁽¹²⁾ The foils were examined in a Siemens Elmiskop Ib operated at 100 kV.

Representative electron micrographs of the alloys are shown in Figs. 7 to 10. The substructure of Mo is the same as that of Ta, so Mo is not shown in the figure. In those alloys that flow plastically, discs were cut from areas near the flowed regions of the crystals in order to observe the dislocation arrangements after slip. A detailed

analysis of the micrographs showed that the following structural effects can be discounted.

1. intermediate phases.
2. Long range ordered regions having dimensions $>300\text{\AA}$, e.g. if the dimension of the ordered region is about three dislocation image widths, i.e. $\approx 300\text{\AA}$, then the dislocations will be expected to be paired. This possibility was investigated by contrast experiments⁽¹³⁾ and unobserved in all of these alloys.
3. small scale precipitates with dimensions $>20\text{\AA}$.
4. interstitial ordering.⁽¹⁴⁻¹⁶⁾

The absence of the latter two effects is not surprising in view of the high purity of the alloys.

In Figs. 7-10 the lines drawn on the micrographs are the projection of the slip vectors $a_0/2 \langle 111 \rangle$ for the given orientations of each micrograph. Ta, Ta-12 Mo, Ta-21 Mo and Mo-7 Ta show numerous dipole loops produced by dislocation interactions. The dislocation debris appears to consist mainly of dipole loops and isolated dislocation segments of strong screw character as indicated by comparison to the projections of the slip vectors. The tendency for the dislocations to lie in straight lines becomes dominant in Ta-21 Mo and Ta-30 Mo. In fact, all the dislocations in Ta-30 Mo are straight and nearly all are screws. This tendency suggests an increase in the Peierls-Nabarro friction with alloying. The micrographs of those crystals which flowed plastically showed that two or more slip systems are operative sometime

after the initial flow.

The only way to determine whether or not localized compositional variations occur over relatively short distances in a solid solution is by careful Kikuchi electron diffraction analysis.⁽¹⁷⁾ If, as the lattice parameter determinations suggest (Fig. 6), there is a tendency for Mo and Ta atoms to separate in adjacent regions without a change in Bravais lattice, then the d-spacings for the same (hkl) reflecting planes in these regions will be slightly different giving rise to paired diffraction spots and Kikuchi lines as discussed elsewhere.⁽¹⁷⁾ This applies to clustering or spinodal decomposition in solid solutions. Since the Kikuchi lines give accurate values of the (magnified) reciprocal lattice vectors, they can be used to determine the d-spacings^(16,17) and to resolve small differences in spacings that would not be detectable by X-rays. Sharp Kikuchi patterns are only obtained from thick regions with a relatively low dislocation density. When the difference in d-spacings of a given set of planes is small ($<0.1\text{\AA}$) pairing of diffraction lines is resolved only for high index reflections and this necessitates the use of a goniometer stage. Such pairing was consistently observed in foils from alloys that were mechanically brittle (Fig. 4). Care was taken to ensure that no small angle boundaries contributed to the pattern, as these could also cause pairing of diffraction lines.⁽¹⁷⁾ Figures 11 and 12 show examples for Ta-50 Mo and Mo-40 Ta from selected areas $\sim 0.3\mu$ diameter.

In Fig. 11 the pairing of the 440 reflections is resolved and the separation of the two lines indicates there is about a 2% difference in d-spacings. From the camera constant, the estimated lattice parameters from regions corresponding to each reflection are $3.26\text{\AA}$ and $3.16\text{\AA}$,

respectively. From Fig. 6 it is seen that the corresponding alloy compositions are about Mo-10 Ta and Ta-34 Mo, i.e. compositions which border those of alloys which cleave in tension.

Figure 12 shows resolved pairing of the h_{22} Kikuchi lines in Mo-40 Ta. Here the difference in d-spacings amounts to about 4% and the corresponding lattice parameters indicate composition differences which are also very close to those found for the Ta-50 Mo alloy.

It may be that the diffuse bands which are sometimes observed in alloys within the composition range Ta-21 Mo to Mo-7 Ta (examples of which are shown in Figs. 8 and 10) actually correspond to regions in which the composition deviates from randomness. The contrast variations in Figs. 8 and 10 can be interpreted in terms of relative electron absorption, in which case the dark regions would be those richest in tantalum. These bands are not extinction contours and do show crystallographic habits. On the other hand such contrast effects could also arise from thickness variations in the foils, e.g. from preferential electrothinning (which might also arise from compositional variations). Thus, it is difficult from the micrographs alone to interpret these effects unambiguously; however, the diffraction evidence shown in Figs. 11 and 12 provides conclusive proof that the brittle alloys are non-random solid solutions and these results serve to emphasize the importance and value of Kikuchi line analyses during electron microscopy investigations.

As a result of demixing in the solid solutions, X-ray diffraction intensities should be different from those expected from random solid solutions. That this is the case is shown in the following.

2.6 X-Ray Intensity Measurements

~~During the lattice parameter work it was observed that the intensity~~ of the (200) line was anomalously large compared to the (110) line in some alloys. From the electron diffraction evidence it is reasonable to propose that this behavior is due to deviations in composition from random solid solution on certain crystallographic planes of the alloy. Such deviations can be expected, since the elastic compliances for both Mo and Ta in and normal to cubic crystal planes are anisotropic (see Table IV) and furthermore the integrated intensity should be affected by crystallographically oriented deviations in composition since the scattering factor of Ta is about twice that of Mo. In order to investigate this possibility a series of X-ray diffraction intensity experiments were carried out.

Since spherical single crystals are the most suitable for making quantitative intensity measurements from two or more sets of reflecting planes in a crystal, a technique was developed for growing such crystals in the zone refiner. (13)

For obtaining accurate intensity measurements, it was desired to set each crystal such that the [001] is coincident with the diffractometer axis. In this setting the two sets of planes (100), (010) and the two sets of (110), ($1\bar{1}0$) and their conjugates can diffract into the detector slit of the counter. In order to make a quantitative comparison between the diffracted intensity of one set of planes with another, the [001] pole must be set to within one half a degree to the diffractometer axis. This required a complete analysis and correction of all sources of alignment errors. The experimental details are described elsewhere. (4)

TABLE IV Ratio of Integrated X-Ray Diffracted Intensities

Alloy	(I_{200}/I_{220})
Mo	2.06
Mo-7 Ta	1.92
Mo-19 Ta	2.63
Mo-28 Ta	3.02
Mo-40 Ta	3.52
Ta-50 Mo	4.43
Ta-42 Mo	1.59
Ta-30 Mo	2.00
Ta-21 Mo	2.05
Ta-12 Mo	2.33
Ta	2.07

Figure 13 shows the results obtained for the Mo-40 Ta alloy. The intensities of the (220) and (200) lines were integrated from peak two minus 0.06° to peak two plus 0.06° . This angular range included about 80% of the area under the intensity versus two theta curve. The purpose of these measurements was to compare the diffracted intensities of (200) and (220) from the same crystal, therefore, the ratio of the integrated intensity of (200) to the integrated intensity of (220) was computed. These ratios are given in Table IV and plotted versus alloy composition in Fig. 14. The line connecting the end points of a binary system corresponds to the intensity ratios that should be observed in a random solid solution. The figure indicates that in the cleavage range of this alloy series there are large deviations from the predicted ideal random solid solution values of (I_{200}/I_{220}) .

3. SUMMARY OF RESULTS

The substructural results and the physical property measurements show that high purity Mo-Ta alloys are all single phase bcc solid solutions. However, all the results indicate that the solid solutions are not ideal, random solutions, e.g. -

1. The lattice parameters exhibit a positive deviation from Vegard's law (Fig. 6).
2. The integrated X-ray intensity ratios (I_{200}/I_{220}) of the alloys show marked deviations from the values expected for random solid solutions, particularly over the composition range corresponding to brittle mechanical behavior (Fig. 14).

3. Kikuchi electron diffraction patterns from alloys which exhibit the largest divergence in (I_{200}/I_{220}) , i.e. Ta-50 Mo and Mo-40 Ta show a difference in lattice parameter which can only be explained by compositional variations in the alloys.
4. Alloys in the range Mo-19 Ta to Mo-58 Ta fail by cleavage on {100} under tensile loading at 273°K.

On the basis of these results a model is proposed which explains the above observations. This is presented and discussed in the following.

4. DISCUSSION

4.1 Structure of the Solid Solutions

The Kikuchi electron diffraction results prove that as the concentration of solute in the alloys increases there is a tendency for one of the two components to be located preferentially on certain planes. The alloy is then divided up into regions or bands which are probably about 2000Å in size. Thus, the results of this investigation suggest that a type of demixing occurs in the solid solutions and which exhibits a crystallographic habit. The characterization of such demixing is explained by the following.

Consider a region of a Ta-50 Mo alloy crystal where the deviation from random solid solution composition occurs on (100). As one samples the distribution of atoms on (100) proceeding in the [100] direction, planes are encountered where the ratio of Ta atoms to Mo atoms is greater than one to one, say nine to one. In a random solid solution the ratio would be one to one. Since the overall composition of the alloy is Ta-50 Mo it is required that on proceeding in the [100] direction (100) planes are encountered where the ratio of Ta to Mo is one to

nine. Thus, one finds $\{100\}$ planes which are rich in Ta and neighboring $\{100\}$ planes which are rich in Mo. A schematic illustration of these quasi-periodic composition deviations as one proceeds in the $[100]$ direction is given in Fig. 15. It is to be noted that the deviations do not have to be periodic, nor does the amplitude of the deviations have to be constant. Furthermore, no ordering, (or spinodal) identity distance is required. The region "a" is rich in tantalum, region "b" is a transition region between "a" and the molybdenum rich region "c". Because a $\{100\}$ plane is equivalent to a $\{010\}$ or $\{001\}$, when deviations exist in a crystal they must generate quasi-cubic domains. One domain will have composition deviations on $\{100\}$, the adjacent on $\{010\}$ and the third adjacent domain on $\{001\}$. The shape of the domain will thus approach a cube. When there are composition deviations on $\{110\}$, then the crystal is divided into six types of domains corresponding to the six equivalent $\{110\}$ planes, and the domain shape approaches a dodecahedron.

The reason for such demixing can be understood from a consideration of the elastic compliances of Mo and Ta crystals which are anisotropic (Table V). It can be argued that in a binary alloy containing elements differing in atomic size, the elements will distribute themselves in such a way so as to maintain the internal strain energy of the crystal at a minimum. In the case of Mo-Ta there is about a 4% atomic size factor difference. When the elastic coefficients are anisotropic, the compliances vary from one crystallographic plane and direction to another. Although no data is available for Mo-Ta alloys, it can be seen from Table V that Ta is most compliant in $\langle 110 \rangle$ and $\langle 112 \rangle$. Thus, one would expect deviations from random solid solution composition on $\{100\}$ in

TABLE V

Elastic compliances under tension parallel to and torsion about some crystallographic directions in Ta and Mo crystals in units of (centimeters)²/(dyne) multiplied by 10¹³.

ℓ_i	Element	$(\frac{1}{E})$ tension in $[hkl]$	$(\frac{1}{G})$ torsion about $[hkl]$
[100]	Ta	6.78	12.0
	Mo	2.8	9.1
[110]	Ta	5.16	8.73
	Mo	3.3	10.1
[111]	Ta	4.67	7.63
	Mo	2.3	10.4
[112]	Ta	5.16	8.73
	Mo	3.3	10.1

From data of refs. 23, 24.

the alloy Ta-12 Mo since Ta atoms are slightly larger than Mo atoms. The observed positive deviation of (I_{200}/I_{220}) from the random solid solution value shown in Fig. 14 is consistent with this argument. Furthermore, it is seen from Table V that a Mo crystal is most compliant in and normal to {110}, thus, in the alloy Mo-7 Ta, deviations from random solid solution composition would be expected on {110}. The observed negative deviation of (I_{200}/I_{220}) from the random solid solution value (Fig. 14) is in agreement with this suggestion. Any conclusion based on an extrapolation of the values given in Table V to higher alloy compositions is considered to be invalid because the X-ray intensity, resistivity and mechanical property data strongly suggest that the elastic coefficients do not vary as a simple linear function of the alloy composition.

The region between the alloy compositions of Ta-50 Mo and Ta-42 Mo in Fig. 14 is of particular interest, since here the elastic coefficients are expected to be changing rapidly with composition. In this region the alloy crystal changes with respect to a random solid solution, from a condition of maximum composition deviation on {100} to a maximum composition deviation on {110}. If the values at Ta-50 Mo and Ta-42 Mo are extreme values, then the composition deviation on {100} is shown by the dashed line in the figure. From this, one would predict that the alloy Ta-44 Mo would correspond to a random solid solution. This alloy would then be expected to flow plastically and have the maximum value of critical resolved shear stress for slip of this alloy series. On the other hand, if the values for Ta-50 Mo and Ta-42 Mo are not the extreme values, then the observed curve will continue to diverge from the random

solid solution line with the result that the line connecting the extreme values approaches the vertical, i.e. some alloy in the range between Ta-50 Mo and Ta-42 Mo corresponds to an inflection point on the graph. Following this latter reasoning leads to an interesting consequence in that the composition at the inflection point could correspond to an alloy crystal which is energetically unstable, because the compliances do not satisfy the restrictions; (19)

$$S_{44} > 0, S_{11} > |S_{12}|, S_{11} + 2S_{12} > 0$$

which is imposed by the fact that the strain energy of a crystal,

$$W = \frac{1}{2} C_{ij} \epsilon_i \epsilon_j$$

must be positive, i.e. greater than zero for all real values of ϵ_{ij} ,

unless all ϵ_{ij} are zero. In the strain energy expression, C_{ij} are the stiffness coefficients and ϵ_{ij} are the strains. A systematic determination of the elastic coefficients of this alloy system is planned to distinguish between the two possibilities.

2 Interpretation of Mechanical Properties

The observed values of the critical resolved shear stress for slip on {110} <111> and for cleavage on {100} as a function of alloy composition are given in Fig. 4. Correlation between the resolved normal stress on the cleavage plane (20) as a function of alloy composition was sought but unobserved, e.g. changes in normal stress of 60 Kg/mm² in nine atomic percent solute increment are considered to be unrealistic. The resolved shear stress on the observed cleavage plane at failure are the values plotted in Fig. 4. It is seen from the figure that the minimum observed cleavage shear stress occurs for Mo-19 Ta and Mo-58 Ta for which the values are about the same. Because of the appearance of slip

lines on those crystals that cleave, some plastic flow also occurs in these alloys.

The probable mechanism responsible for the strengthening as the alloy composition increases, for the crystals which fail by plastic flow will be discussed first. The proposed models of solid solution strengthening predict simple relationships between the critical resolved shear stress τ for slip and the concentration c of solute atoms. (21,22) A plot of $\ln \tau$ against $\ln c$ was made for Ta base alloys from zero to 30 atomic percent Mo, but no simple functional dependence of τ upon c over this range of composition was observed, even when the point at Ta-12 Mo was omitted on the basis that this alloy shows deviations from the random solid solution composition on {100}. It is therefore concluded that current models proposed for solid solution strengthening are not applicable to the Ta-Mo alloy system.

The mechanisms involved in the strengthening of these alloy crystals which flow plastically and the transition to a cleavage mode of failure at high alloy compositions is believed to be the result of a complicated interaction and interdependence between the following:

- (a) The Peierls-Nabarro friction of dislocations, and its variation with alloy composition.
- (b) Deviations from random solid solution composition on {100} in some alloys and on {110} in others.
- (c) Changes in the elastic coefficients upon alloying.
- (d) Changes in the elastic coefficients within regions of the crystals as a result of (b).
- (e) Marginal fulfillment of the strain energy restrictions upon the elastic coefficients due to (b), (c) and (d) above.

Although very little detailed knowledge of the Peierls-Nabarro friction stress is available, (21) this shear stress is exponentially dependent upon the atomic configuration at the core of the dislocation. In crystals where the magnitude and directionality of the interatomic forces are large, the distortion produced by the core of the dislocation would be expected to be restricted to a narrow region and thus the shear stress required to move the dislocation would increase exponentially. Furthermore, such conditions would tend to make the dislocations in a crystal lie in straight lines which correspond to regions in the crystal of minimum core energy. The progressive tendency for dislocations to lie along straight lines is shown by the electron micrographs of Fig. 7 (a-c) as the Mo content of Ta base alloys increases from zero to 30 atomic percent. Such observations strongly suggest that the P-N friction is increasing as the alloy content increases. It is suggested therefore, that the observed, appreciable increase in critical resolved shear stress for slip (Fig. 4) is the result of a marked increase in the P-N friction of dislocations.

In those alloys which are non-random solutions, dislocations should be attracted to those regions of lower shear modulus and repelled from regions of higher shear modulus, i.e. the energies of dislocations will be different in Ta-rich than Mo-rich regions.

Compositional demixing on {100} and {110} as well as changes in the elastic coefficients - which are directly related to the magnitude and directionality of the interatomic forces - both change the P-N friction stress. Changes in the elastic coefficients will result from both compositional deviations and simple alloying. (2,23,24) The variation

of the electrical resistivity as a function of alloy composition shown in Fig. 2 indicates that there is a marked change in the density of states in the d and s bands⁽⁴⁾ and/or in the elastic coefficients near the compositions Mo-20 Ta, Mo-50 Ta and Ta-12 Mo since there are marked changes in slope at these compositions. The changes in the elastic coefficients which will have the greatest effect on the P-N friction are those changes in S_{44} , S_{11} and S_{12} which produce the greatest deviation in the isotropic condition for the compliances $S_{44} = 2(S_{11} - S_{12})$ or as written for the stiffnesses $C_{44} = \frac{C_{11} - C_{12}}{2}$. From an intuitive physical argument, composition deviations on {100} and {110} are interdependent with the elastic coefficients, e.g. from Fig. 14 the Ta {100} are the most compliant thus the alloy crystal Ta-12 Mo exhibits a composition deviation such that there are Ta-rich and Mo-rich regions on {100} which would not exist in a random solid solution. Because of this, the compliance in and normal to {100} in Ta-12 Mo will change. In order to verify the above concepts, the elastic coefficients as a function of alloy compositions are required.

The previous models that have been proposed for the mechanisms of cleavage⁽²¹⁾ invoke the stress normal to the cleavage plane as being the determining parameter. The normal stress shows no obvious significance in the cleavage of the alloys investigated. However, as can be seen from Fig. 4 there is an increase in the shear stress on the cleavage plane as the alloy composition increases or decreases from Mo-28 Ta. One can reason that the minimum cleavage shear stress will occur when the elastic shear strain energy is at a maximum at some region in the crystal. In a Ta-Mo alloy crystal one would expect the maximum shear

strain energy to be attained in regions which possess the largest internal strain energy. When there exists deviations from random solid solution compositions on {100} the maximum shear strain energy will be attained in the Ta-rich, Mo-rich or transition regions which lie parallel to the cleavage plane. In the Ta-42 Mo crystal which exhibits deviations on {110}, the intersection of {110} and {1 $\bar{1}$ 0} domains generate a transition region which lies parallel to the {010} plane. This is a likely region where changes in C_{ij} are occurring and where the internal shear strain energy attains a maximum.

Because slip lines are observed on those crystals that cleave and that the cleavage appears to be dependent on shear stress, the detailed mechanism of cleavage in this alloy system must include the influence of dislocations. The fact that the presence of dislocations in a crystal can change the magnitude of the elastic coefficients in local regions of the crystal has been recognized and discussed.⁽²¹⁾ The proposed mechanism for cleavage in the Ta-Mo alloy system is as follows: In the range of alloy compositions which exhibits cleavage the values of the elastic compliances are such that the restriction imposed on the compliances by a positive strain energy of a crystal are only marginally fulfilled. These restrictions are:⁽¹⁹⁾

- a) $S_{44} > 0$
- b) $S_{11} > |S_{12}|$
- c) $S_{11} + 2S_{12} > 0$

Substitution of the values for pure Ta, i.e. $S_{11} = 6.78 \times 10^{-13} \text{ cm}^2/\text{d}$, $S_{12} = 2.47 \times 10^{-13} \text{ cm}^2/\text{d}$ and $S_{44} = 11.93 \times 10^{-13} \text{ cm}^2/\text{d}$, shows that an increase in $|S_{12}|$ of 0.92 is sufficient to violate restriction (c). The same

analysis using values for Mo show that an increase* in the $|S_{12}|$ of only 0.62 is sufficient to violate restriction (c). Therefore, it is probable that under the influence of alloying and the associated composition deviations the restrictions on the compliances, which are the same as on the stiffnesses, are just marginally satisfied in those alloys which cleave. The role of dislocations in this mechanism is to alter the elastic compliances to values which no longer satisfy the above restrictions in regions of the crystal where the dislocation density has achieved some critical value. The crystal then becomes energetically unstable and it fails along a crystallographic set of planes which releases a maximum amount of strain energy. Due to the lack of a detailed knowledge of the elastic coefficients of these alloy crystals, the proposed mechanism cannot be tested in a quantitative manner. However, similar arguments can be applied to the other bcc alloys shown in Table I using the data of Table VI.

The behavior of the lattice parameter as a function of alloy composition, Fig. 6, exhibits no marked anomalies. Beyond the composition Mo-7 Ta a positive deviation from Vegard's law is observed. The maximum deviation occurs near the alloy compositions Mo-33 Ta (minimum cleavage shear stress) and the second largest deviation occurs near Ta-30 Mo (maximum shear stress for slip). The purpose of measuring the lattice parameters of the alloy series was to determine if there existed any inflections in the lattice parameter versus % solute for some particular

* It is realized that a change in one elastic coefficient is necessarily accompanied by changes in the others. However, the magnitude of the change need not be the same for all the coefficients. Hence, changes in the coefficients can occur which will reduce the margin of fulfillment of the strain energy restrictions.

TABLE VI

Element	S_{11}	$-S_{12}$	S_{44}	$(S_{11} - S_{12})^*$	$(S_{11} + 2S_{12})^*$
Nb	6.60	2.33	35.0	4.27	1.94
Ta	6.78	2.47	11.98	4.31	1.84
Mo	2.8	0.78	9.1	2.02	1.24
W	2.57	0.729	6.60	1.84	1.11
<p>* A measure of fulfillment of elastic strain energy restrictions, i.e. $S_{11} > S_{12}$, $S_{11} + 2S_{12} > 0$ and $S_{44} > 0$.</p>					

From the data of refs. 23-25.

alloy composition. As can be seen from the figure, the maximum positive deviation from Vegard's law at Mo-33 Ta and Ta-30 Mo suggests clustering phenomena which are consistent with the model of composition deviations derived from the Kikuchi diffraction patterns.

Since X-ray measurements are obtained from relatively large volumes of a crystal which could contain many differently oriented compositional "bands", local variations in lattice parameter from one band to another are difficult to resolve. The X-ray results thus indicate cubic symmetry and the lattice parameters are only average values for the volume of crystal irradiated.

The advantage of Kikuchi electron diffraction from selected areas is that local compositional variations can be recognized. The diffraction evidence from Figs. 11 and 12 provides conclusive proof that the proposed compositional variations do, in fact, exist in this alloy system.

It is interesting to compare the present results to the known mechanical behavior of the other bcc solid solutions listed in Table I. (1-3) The values of the elastic coefficients of the four elements of interest are given in Table VII. It is immediately apparent from these tables that a correlation exists between the mechanical behavior of the alloys and the elastic coefficients of the respective components. Those alloys that are brittle are composed of elements of widely different elastic constants (Ta-Mo, Nb-W, Nb-Mo, Ta-W) whereas alloys that are ductile (Nb-Ta, Mo-W) contain elements with similar elastic coefficients. By analogy to the present results on Ta-Mo, non-random solid solutions are to be expected in the brittle ranges of compositions in those alloys whose components have elastic coefficients which are not only quite

TABLE VII

Elastic compliances under tension parallel to and
torsion about some crystallographic directions in
units of centimeters/dyne multiplied by 10^{13}

Element	ℓ_i	$\frac{1}{E}$ tension in $[hkl]$	$\frac{1}{G}$ torsion about $[hkl]$
Ta	[100]	6.78	12.0
	[110]	5.16	8.73
	[111]	4.67	7.67
	[112]	5.16	8.76
Mo	[100]	2.8	9.1
	[110]	3.3	10.1
	[111]	2.3	10.4
	[112]	3.3	10.1
Nb	[100]	6.60	35.0
	[110]	10.85	43.5
	[111]	12.32	46.4
	[112]	10.85	43.5
W	[100]	2.57	6.60
	[110]	2.57	6.60
	[111]	2.57	6.60
	[112]	2.57	6.60

different but which are also anisotropic. Thus, a knowledge of these coefficients and how they change on alloying enables a prediction to be made of the mechanical properties expected when alloys are formed by various combinations of bcc elements of groups V and VI.

6. CONCLUSIONS

1. The Ta-Mo alloy single crystals cleave under tensile loads in the composition range Mo-19 Ta to Mo-58 Ta on {100} planes at 273°K.
2. The critical resolved shear stress for plastic flow is a non-linear function of alloy composition. The increase in the shear stress for flow is the result of an increase in the Peierls-Nabarro friction of a dislocation which occurs through composition deviations on {100} or {110}, alloying, and changes in the elastic coefficients.
3. The Ta-Mo alloy series has a bcc crystal structure showing no long range order with dimension $> 300\text{\AA}$, nor intermediate phases.
4. The lattice parameter versus alloy composition shows a positive deviation from Vegard's law for all compositions from Mo-18 Ta to pure Ta and is interpreted as evidence for demixing.
5. Kikuchi electron diffraction patterns prove that the composition deviates from randomness for alloys in the range Mo-19 Ta to Mo-50 Ta. These alloys show cleavage at 273°K which appears to be dependent on the shear stress on the cleavage plane {100}.
6. The proposed mechanism of cleavage is as follows. Cleavage occurs due to a violation of the strain energy restrictions on the elastic coefficients in a local region of the crystal. Alloying and the existence of crystallographic composition deviations change the elastic coefficients, in the range of alloys which cleave, to values which marginally fulfill

the restrictions imposed on them by the necessity of a positive definite strain energy. The presence of a critical number of dislocations in a local region produces changes in the values of the elastic coefficients such that the strain energy restrictions are violated in this region. The crystal is then energetically unstable and cleaves on the crystallographic plane which releases the maximum strain energy.

7. Marked changes in the slope of ($\rho_{273^\circ\text{K}} - \rho_{4.2^\circ\text{K}}$) versus alloy composition at Mo-20 Ta, Mo-50 Ta and Ta-12 Mo suggest that there is a relatively large change occurring in the density of states in the d and s bands and/or marked changes in the elastic coefficients at these compositions.

8. Brittle behavior is expected in solid solutions formed between elements whose elastic compliances are very different.

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Tables

1. Part of the periodic table showing data for Nb, Mo, Ta and W; the lower scheme shows mechanical behavior of alloys of 1:1 atomic ratio.
2. Specific resistivities at three temperatures and the lattice parameters at 23°C for the Ta-Mo single crystals.
3. Calculated shear stresses for plastic flow or cleavage.
4. Ratios of experimentally determined X-ray diffracted intensities.
5. The elastic compliances under tension parallel to, and torsion about, crystallographic directions in Ta and Mo calculated from data in refs. 23 and 24. Values in $\text{cm}^2/\text{dyne} \times 10^{13}$.
6. Elastic compliances calculated from data of refs. 23-25. Values in $\text{cm}^2/\text{dyne} \times 10^{13}$.
7. The elastic compliances under tension parallel to, and torsion about, crystallographic directions for Ta, Mo, Nb and W. Values in $\text{cm}^2/\text{dyne} \times 10^{13}$.

Figure Captions

- Figure 1 Specific resistivities vs. alloy compositions for Mo-Ta single crystals.
- Figure 2 Values of $(\rho_{273^\circ\text{K}} - \rho_{4.2^\circ\text{K}})$ vs. alloy composition for Mo-Ta single crystals.
- Figure 3 (a) Orientations of the Mo-Ta single crystals grown by zone refining.
(b) Load-elongation curves and orientations of alloy single crystals which flowed plastically at 273°K . The strain rate was $3.33 \times 10^{-4}/\text{sec}$.
- Figure 4 Plots of the critical shear stress for plastic flow or cleavage vs. alloy composition.
- Figure 5 X-ray intensity of the $\{211\}$ line of Ta-42 Mo single crystal.
- Figure 6 Lattice parameters of Ta-Mo alloys obtained from non-annealed filed powders. Squares are from ref. 10, the dotted line is a plot of Vegard's Law from data of ref. 1 and the solid line is from present data. Circles represent maximum error in experimental measurements.
- Figure 7 (a,b,c) Electron micrographs showing dislocation arrangements in alloys which flowed plastically. Most of the debris is in the form of dipoles. Slip directions are indicated. Notice straight dislocations (probably screws) in (c). Foil orientations are a) $\sim [210]$, b) $\sim [430]$, c) $\sim [311]$.
- Figure 8 Electron micrograph of alloy Ta-12 Mo showing diffuse banded structure. Orientation $\sim [311]$, bands parallel to $\{001\}$.

The projected width of the (100) bands are larger than those on (001) and (010). Band width $\sim 2000\text{\AA}$.

Figure 9 (a-e) Electron micrographs from alloys which cleaved, showing a very low dislocation density. In (b) the cleavage edge parallel to (001) is shown. Foil orientations are a) $\sim [711]$, b) $\sim [110]$, c) $\sim [210]$, d) $\sim [310]$, e) $\sim [311]$.

Figure 10 Banded structure in Mo-7 Ta. Orientation $\sim [210]$, bands parallel to (110).

Figure 11 Electron diffraction pattern of Ta-50 Mo alloy showing pairing of the (440) Kikuchi lines as a result of the contribution of two lattice parameters corresponding to a Ta-rich and a Mo-rich region in the crystal. Orientation $\sim [001]$.

Figure 12 Similar to Fig. 11, showing pairing of the 422 Kikuchi reflections in Mo-40 Ta. Foil orientation $\sim [11\bar{3}]$.

Figure 13 X-ray diffracted intensity for 200 and 220 lines of Mo-40 Ta crystals.

Figure 14 Plot of the X-ray integrated intensity ratios (I_{200}/I_{220}) vs. alloy composition for Mo-Ta single crystals. The solid line represents calculated ratios for random solid solutions.

Figure 15 Scheme showing composition variations corresponding to the observed demixing of solid solutions. The ratios are assumed to deviate from 1:1 as shown.

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MUB-4442

Fig. 1

MUB-4443

Fig. 2

Fig. 3a

MUB-4455

Fig. 3b

MUB-4447

Fig. 4

MUB-4433

Fig. 5

MUB-6626

Fig. 6

ZN-5003

Fig. 7a b c

Ta-12 Mo

ZN-5014

Fig. 8

ZN-5004

Fig. 9a-e

Mo-7Ta

ZN-5015

Fig. 10

$\overset{\bullet}{350}$ $\overset{\bullet}{440}$

$\overset{\bullet}{240}$ $\overset{\bullet}{330}$

$\overset{\bullet}{130}$ $\overset{\bullet}{220}$

$\overset{\bullet}{020}$ $\overset{\bullet}{110}$

$\overset{\bullet}{\bar{1}10}$ $\overset{\bullet}{000}$

ZN-5016

Fig. 11

422

121

211

$\bar{1}10$

000

ZN 5017

Fig. 12

MUB-6627

Fig. 13

MUB-4452

Fig. 14

MUB-6628

Fig. 15

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