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THE INFLUENCE OF MICROSTRUCTURE ON FRACTURE OF DRAWN TUNGSTEN WIRE

by

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SUMMARY

The microstructures and longitudinal fracture resistances of lamp-doped and undoped tungsten wire were examined in the as-drawn condition and after annealing at 600 °C to 1500 °C A variety of experimental techniques were employed including Auger Electron Spectroscopy, Scanning Electron Microscopy, Transmission Electron Microscopy and a newly developed mechanical testing technique The longitudinal fracture mode was intergranular for all wires and a second phase was observed on the grain boundaries of all doped wires. High concentrations of the dopant element potassium were present on the fracture surfaces of doped wires and experimental evidence was obtained which suggests they may be due to post-fracture surface diffusion. Doped wires demonstrated increasing amounts of structure coarsening up to 1500 °C whereas large equi-axed grains were formed in undoped wires annealed at 1300 °C and 1500 °C The longitudinal fracture resistance of undoped wire was unaltered by annealing at 1050 °C and below, but decreases dramatically after annealing at 1300 °C and 1500 °C. In contrast, the fracture resistance of doped wire decreased after annealing at 1050 °C and 1300 °C, but increased after annealing at 1500 °C Fracture resistance is discussed in terms of microstructure and fracture surface chemistry

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KEY WORDS

fracture, tungsten, Auger Electron Spectroscopy, grain boundaries, embrittlement

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1. INTRODUCTION

Heavily drawn tungsten wire displays considerable ductility under a variety of deformation processing methods such as drawing and coiling However, these wires are susceptible to brittle separation along longitudinal grain boundaries. Longitudinal intergranular cracks may develop when the stress component normal to the wire axis is predominent and exceeds certain threshold levels For example, failure of wires under simple shearing conditions is invariably accompanied by longitudinal intergranular cracking and wires with surface flaws separate longitudinally along grain boundaries when they are simultaneously bent and stretched.[1] Varga has estimated that fiber boundary strength is only about 14% of wire tensile strength.[2] However, quantitative information on the susceptibility of wires to intergranular fracture is not abundant and the influence of metallurgical parameters on fracture resistance is unknown.

The present work was undertaken to investigate the influence of microstructural parameters such as grain size, second phases and grain boundary chemistry on the resistance of drawn tungsten wire to longitudinal intergranular separation. This was accomplished by observing the influence of the microstructural changes produced by low temperature annealing on the fracture resistance of lamp-doped and undoped tungsten wires. A newly developed mechanical testing technique was employed to determine the fracture resistance of wires, and fracture surface chemistry and microstructural details were determined with a Scanning Auger Microprobe (SAM), Scanning Electron Microscope (SEM) and Transmission Electron Microscope (TEM), respectively. Both undoped and commercially lamp-doped wires were examined since their microstructural responses to annealing treatments are markedly different. Lamp-doped wire contains three dopant elements (K, Al, and Si) however, a variety of experimental techniques (TEM,[3] AES,[4] SIMS[5]) have established that K is the key dopant element and is responsible for the formation of rows of bubbles or voids parallel to the wire axis during annealing These bubble rows restrict boundary motion and lead to the

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development of a unique creep resistant grain structure at elevated temperatures.[6,7]

2. EXPERIMENTAL PROCEDURE AND RESULTS

2.1 Materials

The materials used in this investigation were 0.635mm diameter General Electric lamp-doped tungsten wire (type 218) and similarly processed undoped wire Both wires had been swaged and drawn to a true strain of 5.1 in accordance with the standard procedures used in tungsten wire production [8] Sections of wire were cleaned in boiling hydrogen peroxide, placed in quartz tubes which were baked out at 300°C, evacuated to a pressure of about 1 3 x 10^{-4} Pa, and sealed. The samples were placed in a furnace preheated to either 600°C, 800°C, 1050°C, 1300°C, or 1500°C for two hours and water quenched. In addition to annealed samples, as received samples were also studied

2 2 Fractography

The longitudinal fracture surfaces of all wires were examined using Scanning Electron Microscopy in order to establish the mode of fracture and the microstructural changes which had occurred during annealing. In addition, single stage carbon replicas of the fracture surfaces of as-received doped wire and doped wire annealed at 600°C and 800°C were examined by TEM The fracture mode was intergranular for all wires.

As-received, doped and undoped wires both had highly-elongated fibrous grain structures (Figure 1a) and the average grain width of both was estimated to be 0.4μm from SEM fractographs. When the grain boundaries of doped wire were examined at very high magnification using TEM replica fractography, highly elongated second phase features oriented parallel to the wire axis were observed (Figure 1b). The features were 100-200Å wide, several thousand Å long, and covered a very small area fraction (<1%) of the fracture surface.

SEM fractography of wires annealed at 600°C and 800°C showed that no apparent changes had occurred in grain size or morphology. TEM replica fractography of doped wire annealed at 600°C revealed second phase features identical to those observed in as received wire. However, after the 800°C anneal the elongated second phase had increased in width and in some cases had divided into rows of discreet spherical voids (Figure 1c).

Grain widths increased and transverse grain boundaries were observed for both doped and undoped wires annealed at 1050°C (Figure 1d). The extent of both of these events was greater in undoped wire. Rows of spherical voids about 2000Å in diameter could be resolved on the grain boundaries of doped wire using SEM.

Annealing doped wires at 1300°C and 1500°C results in significant grain broadening and the formation of many new transverse boundaries (Figure 3e). Both changes occurred to a much greater extent at 1500°C. As for doped wire annealed at 1050°C, rows of voids or bubbles about 2000Å in diameter (Figure 1e) were observed. The elongated grains in undoped wire became large equiaxed grains during the 1300°C and 1500°C anneals (Figure 1f).

2 3 Fracture Surface Chemistry

The spectrometer used to investigate the chemistry of interfibrous fracture surfaces of the wires was a Physical Electronic (PHI) Model 545 Scanning Auger Microprobe (SAM) which permitted low resolution secondary electron images of the fracture surfaces. In situ fracture of specimens was done with the PHI Model 10-520 specimen breaker. Since the specimen breaker provided for bending only, a method of wire notching was developed which allowed longitudinal fracture of specimens (Figure 2). In this method a diamond wafering blade was used to cut diametrically opposed notches about 0.4mm deep and 0.3mm apart. The notched wire was then placed into a notched copper tube which was crushed to hold the wire stationary and mounted into the specimen stage. In most cases, two samples of each wire condition were prepared.

At spectrometer pressures of the order of 10^{-8}Pa, the specimen breaker was used to bend the copper tube 90° at its notch which fractured the tungsten wire longitudinally between its notches and oriented the fracture surface perpendicular to the wire axis. The fracture surface was then rotated before the analyzer For all of the Auger analyses, the primary electron beam voltage was 3kV corresponding to a beam diameter of ~25μm and typical sample currents were 100 to 150nA.

Oxygen and carbon were present in relatively small amounts (<3at.%) on many of the fracture surfaces of doped and undoped wires. Their concentrations, however, did not vary in any systematic manner with annealing temperature or wire type and increased as a function of time. Based on these observations and because both oxygen and carbon are common spectrometer contaminants, it is likely that both elements were being adsorbed

after sample fracture. Accordingly, intrinsic grain boundary concentrations of carbon and oxygen could not be obtained.

Significant levels of potassium were found on the interfibrous fracture surfaces of doped wires. Figure 3 shows a typical spectrum for as-received material. In Figure 4a, the ratio of the peak-to-peak height of the potassium Auger peak (252eV) to tungsten Auger peaks (169eV manifold) has been plotted as a function of annealing temperature. The Auger peak-to-peak heights have been demonstrated to be directly proportional to surface atomic concentrations.[9] The atomic fraction of potassium was estimated using techniques outlined in the Handbook of Auger Electron Spectroscopy.[10] Results are displayed on the right hand axis of Figure 4a.

After taking at least four selected area Auger analyses on the fracture surface, a representative area was chosen to obtain chemical depth distributions by argon ion sputtering. The method used for depth profiling was to ion mill for time intervals of 15 to 60 seconds followed by obtaining a full spectrum. When the profile data were plotted as the raw peak-to-peak heights vs. ion milling time, two types of profiles were obtained. For as-drawn wire and wire annealed at 600°C, extensive sputtering times (~600 sec) were required for potassium removal (Figure 4b) whereas for wires annealed at 800°C and above, relatively short sputtering times (~30 sec) were required (Figure 4c). In a separate measurement, the material removal rate under identical ion beam conditions was determined to be 20Å/min. for pure tungsten vapor deposited onto a glass slide. Based on this measurement, approximate sputter depths are given on the top axis of Figures 4b and c.

2.4 Mechanical Testing

A new mechanical testing technique was developed and used to determine the relative resistance of wires to longitudinal fracture. In this test, the wire is compressed between the edges of two tungsten carbide tool bits of square cross section (Figure 5a). The axis of the wire is oriented normal to the edges of the tool bits which have a radius of 0.13cm. When the load applied at a cross head speed of 4.2×10^{-6} cm/sec. with a screw driven Instron Machine reached a critical value, wires delaminated and a sharp load decrease occurred. This critical laod indicates resistance to fracture and will be referred to as the delamination laod. An example of wire fractured by the indentation test is shown in Figure 5b. Twenty tests were made on each wire type and the mean delamination load and the standard deviation of the mean were computed. The dependence of delamination load on

annealing temperature is presented in Figure 6 for both doped and undoped wires.

3. DISCUSSION

3.1 Microstructures

The as-drawn structures of doped and undoped wires consisted of highly elongated grains about 0.4μm wide. Other investigations have shown that these grains are ribbon-like in shape and contain several subgrains or cells.[6, 11] The highly elongated second phase present on grain boundaries of doped wires is identical in morphology to that observed by other investigators.[6, 7] Although the composition of the second phase has not been determined directly, it is generally believed to result from the deformation of potassium containing pores present in the precursor powder metallurgy ingot.[6, 7, 12]

Grain shape and size, as determined with SEM fractography, were unaltered during the 600°C and 800°C anneals of doped and undoped wires. Recovery processes, however, such as the removal of excess vacancies, dislocation relaxation and polygonization, are known to take place in tungsten at these temperatures.[13] The elongated second phase features present in as-drawn wire increased in diameter and were replaced in some instances by rows of discreet bubbles during annealing in 800°C. Snow[6] has noted similar morphological changes at 800°C in doped tungsten and has identified elemental potassium in the bubbles. The process of bubble formation has been termed spheriodization[6] or ovulation[7] and its mechanisms have been discussed elsewhere. Warlimont has identified a second family of bubble not aligned in rows on grain boundaries in annealed doped wire and has speculated that they are the result of nucleation rather than spheriodization.[12] No evidence for the second type of bubble was obtained in the present investigation after any annealing treatment.

The grain sizes and morphologies of doped and undoped wires were altered by the 1050°C, 1300°C, and 1500°C anneals. Grains broadened and new transverse boundaries were created during the 1050°C anneal of undoped wire. While the same events occurred in doped wire, they proceeded to a much lesser degree. The fibrous structure of undoped wires was completely eliminated during the 1300°C and 1500°C anneals and was replaced by relatively large equiaxed grains. As annealing temperature was increased to 1300°C and 1500°C for doped wire, progressively more grain broadening occurred. Rows of large

(2000Å) bubbles were present on the fiber boundaries after the 1050°C, 1300°C and 1500° anneals. It was not evident whether these bubbles had been formed by the expansion of those bubbles observed after the 800°C anneal or resulted from boundary interactions with new bubbles. Numerous investigators have found that the bubbles formed in doped tungsten strongly inhibit boundary migration.[6, 7, 12] In the present study, bubbles appear to have been responsible for the retention of elongated grains in doped wires heated to 1300°C and 1500°C.

3.2 Fracture Surface Chemistry

Second phase features were present on the grain boundaries of doped wires in as-received condition and after all annealing treatments employed. These undoubtly contain potassium since potassium was the only element identified in significant quantitites on fractures surfaces except for small amounts of O and C which may have been adsorbed from the spectrometer. These second phase features, however, covered only small area fractions of fracture surfaces, typically <1%, especially for as-received wire and wires annealed at temperatures below 1050°C. Therefore, the fracture surface potassium concentrations observed, as high as 20 at.% for some cases, could not be readily accounted for by the presence of second phases. High potassium levels would be observed if, in addition to the potassium containing bubbles, a potassium layer existed on the fracture surface of thickness less than the Auger electron escape depth of tungsten or 7 - 17A.

Provided primary beam-specimen interactions can be rule out, factors that could have contributed to such a layer are, one, prefracture grain boundary potassium, and two, post fracture potassium surface diffusion from second phase potassium intersected by the fracture path. Potassium is indeed known to diffuse readily on the surface of tungsten at room temperature.[15] Calculations based on information in reference 15 suggest that the mean diffusion distance in the typical 1000 second interval between sample fracture and Auger analysis is about 1μm. Since this distance is on the order of the spacing of second phase potassium containing particles, it is a real possibility that surface diffusion had contributed to the high fracture surface potassium concentrations observed.

Possible potassium surface diffusion after sample fracture made the interpretation of Auger results (Figures 4a and b) difficult. Therefore,

additional experiments were conducted to clarify the potential influence of potassium surface diffusion on fracture surface chemistry. Two identical samples of 2.03mm diameter heavily worked and doped tungsten rod were used and notched perpendicular to their axes to initiate transgranular cleavage. In situ fracture Auger analyses were done on both samples using the same experimental conditions as for the 0.635mm wires. However, prior to fracture, one sample was contact cooled by circulating liquid nitrogen through the specimen breaker to an equilibrium temperature of -160°C.

High potassium concentrations were present on fracture surfaces of the room temperature and pre-cooled samples. In fact, the peak-to-peak ratio of the potassium (252eV) to tungsten (169eV manifold) was greater than that for the intergranular fracture surfaces of the 0.635mm wires. The chemical profile data for both samples was similar to that for samples annealed to 800°C (Figure 4a). That is, a very rapid decrease was observed for potassium within a sputtered depth of ~10Å. Subsequent SEM fractography demonstrated that both samples fractured predominantly by transgranular cleavage with only small areas of intergranular separation (Figure 7).

These results indicate that potassium surface diffusion had occurred from elemental potassium contained in voids or from the localized intergranular areas on the fracture surfaces. Sample cooling prior to fracture was apparently ineffective in preventing diffusion. Results also suggest that surface diffusion may play a dominant role in determining the potassium concentration observed on intergranular fracture surfaces in this and other studies.[4] The rapid removal of potassium from the intergranular fracture surfaces indicated that the surface layers formed by diffusion were very thin.

The relative contribution of potassium surface diffusion to fracture surface chemistries cannot be evaluated at this time and a detailed interpretation of the results in Figure 4a will not be attempted. However, potassium surface diffusion layers should be very thin and, hence, rapidly removed by ion sputtering. The depth distributions of potassium for the as-received wire and wire annealed at 600°C show that the fracture surface potassium is not due solely to surface diffusion but rather a possible combination of surface diffusion and a relatively deep zone of potassium enrichment. Warlimont has speculated the existence of such a potassium rich boundary zone which is created by the collapse and incorporation of potassium bubbles into the boundary during material working.[12]

3.3 Delamination Resistance

The susceptibility of tungsten to intergranular fracture has often been attributed to the segregation of interstitials to grain boundaries.[15,16] As noted previously, relatively small concentrations of both oxygen and carbon were observed on some intergranular fracture surfaces but these concentrations did not very in any systematic manner with sample type of heat treatment. Unfortunately, the evidence indicates that adsorption had contributed to the concentrations observed. Due to the random nature of the concentrations, no correlation with delamination laod could be established. Since neither oxygen nor carbon was found on some intergranular fracture surfaces, it may be concluded that brittle intergranular fracture does not require significant concentrations of either element.

No direct correlation was established between either fracture surface potassium concentration and delamination resistance or potassium depth distribution and delamination resistance. The delamination loads of doped and undoped as-drawn wires were comparable, although high potassium concentrations were present on the intergranular fracture surfaces of doped wires. Annealing doped wires at 800°C did not alter delamination resistance despite large reductions in both fracture surface potassium concentration and its persistence during sputter etching. The lack of correlation between potassium concentration and delamination load implies that either other factors control delaminationload or that true boundary potassium concentrations were being masked by potassium surface diffusion. Based on the inability to determine true prefracture grain boundary concentrations, changes in delamination load of doped wires have been rationalized in terms of microstructure. It is assumed that the principle influence of potassium is to modify microstructural responses to annealing and, therefore, indirectly controlled delamination load.

Except for a slight increase after the 800°C anneal, the delamination load of undoped wires was not significantly changed by annealing at 1050°C or below. Therefore, the recovery process known to be active in tungsten at these temperatures and the fiber broadening which occurred at 1050° did not exert a strong influence on wire delamination resistance. The large decrease in delamination load for wires annealed at 1300°C and 1500°C can definitely be attributed to the development of large equiaxed grains.

The delamination resistance of as received doped and undoped wires were comparable, indicating the elongated second phase in doped wires did not influence strength. As for undoped wire, the delamination resistance

of doped wire was not significantly altered by the 600°C and 800°C anneals. This indicates the second phase morphological changes which occurred at 800°C (bubble row formation) and recovery processes which are active in tungsten at this temperature did not effect delamination resistance. After annealing at 1050°C and 1300°C, the delamination resistance of doped wire was dramatically reduced. Since fiber size and shape in these wires were similar to that of undoped wire annealed at 1050°C (high delamination resistance) the delamination level decrease in doped wire must be due to changes unique to doped wire. The rows of relatively large bubbles (2000Å) present on fiber boundaries may have acted to concentrate stress and lower wire delamination resistance. In an analogous situation, the presence of thoria particles in thoriated tungsten is often blamed for poor material fabricability due to wire delamination. The high delamination resistance of doped wire annealed at 1500°C contradicts the above rationalization since large bubbles were also present. However, the extent of structure coarsening and transverse boundary development in this wire was much greater and these changes resulted in a much more tortuous (less linear) path for crack propagation.

4. Summary and Conclusions

1. The longitudinal fracture mode of doped and undoped tungsten wires was intergranular in as-drawn condition and after two hour vacuum anneals at either 600°C, 800°C, 1050°C, 1300°C or 1500°C

2. A second phase was present on the grain bounaries of as-drawn doped wire and after all annealing treatments employed. This second phase was thin and highly elongated in as-drawn wire and spherodized into bubble rows at 800°C and above.

3. High potassium concentrations were present on the interfibrous fracture surfaces of as-drawn doped wire and after all annealing treatments employed. Annealing temperatures of 800°C and above reduced the sputter etching times required for the removal of potassium from fracture surfaces. The presence of high potassium concentrations on transgranular fracture surfaces of doped tungsten rods indicates post-fracture potassium surface diffusion occurs and may have influenced the Auger analysis of intergranular fracture surfaces.

4. No direct correlation was observed between fracture surface potassium concentrations and delamination resistance.

5. The delamination resistance of undoped wire was insensitive to annealing temperature provided a fibrous microstructure was retained. However, at 1300°C and 1500°C large equiaxed grains were formed and delamination load was reduced.

6. Although the fibrous structure of doped wire was retained at all annealing temperatures, delamination load was sensitive to annealing temperature and was markedly reduced by 1050°C and 1300°C anneals. This reduction was unique to the doped wire.

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APPENDIX: Additional Experimental Data on Indentation (Compression) Testing

Introductory Remarks

The indentation testing method has been developed primarily as a laboratory tool to determine on a comparative basis the susceptibility of tungsten wires to split failure. The test is accomplished by indenting two symmetrical anvils on a tungsten wire which produces the necessary transverse tensile stress to cause splitting of fibrous materials All the tests have been made in an Instron Machine where the load increases with increasing displacements of the cross head. When the split occurs, the applied load drops suddenly; this peak load has been used as an indication of the resistance of wires to split failure.

While the indentation test gives an indication of split tendency, the peak load cannot be readily translated into a more meaningful quantity, such as the stress, strain, or stress intensity For this reason, effects of mandrel geometry and wire diameter on split tendencies have been examined Experimental results are summarized in the following

Experimental Methods and Results

Since it is desirable to establish the split tendency in terms of variations in wire diameter (or in terms of processing history), a procedure must be determined which forms a basis for testing wires with different diameters. One of the important parameters that determines the split load is the ratio of indentor radius to wire diameter, r/d, as defined in Figure A-1 Different ratios of r/d that have been examined are summarized in Table A-I. These different combinations of indentor radius to wire diameter have been selected so that the ratio, $\frac{r}{d}$, remains the same for different indentor radius-wire diameter combinations, as indicated by the dashed lines

Results of test data obtained with wires with oxide (Wire #1738) and three different diameters are summarized in Table A-II. The general trend is that the split load increases as the ratio, r/d, increases. The split load also increases with increasing r and increasing d for constant d and r, respectively It might be pointed out that the surface oxide reduces the split load (Table A-II and Figure 6 in the main text).

The effect of indentor radius on split load vs annealing temperature relationship is shown in Figure A-2 The general trend is consistent with the test data (Figure 6) obtained with r/d = 2 3.

Some of the split tungsten wires are shown in Figure A-3. An early stage of crack initiation next to the indentor mark is shown in Figure A-3(a) for the doped wire tested in the as-received condition. An early stage and a fully developed cracks are shown in Figure A-3(b) and (c) for the doped wire tested after an annealing treatment at 1050^{o}C for 2 hours.

Table A-I, A Summary of $(\frac{r}{d})$ Ratios Tested

Wire diameter, in.	Indentor radius, in	r_1=0.005	r_2=0.017	r_3=0.055	r_4=∞
d_1=0.007		0.71	2.43	7.85	--
d_2=0.025		0.20	0.68	2.3	--
d_3=0.082			0.21	0.69	--

TABLE A-I1

A Summary of Split Load for Different Combinations of r/d, Oxide Present

Wire diameter, in	Indentor radius, in	r_1=0.05	r_2=0.017	r_3=0.055	r_4=∞
d_1=0.007		12	8	10	--
d_2=0.025		--	34	50	270
d_3=0.082		--	--	148	--

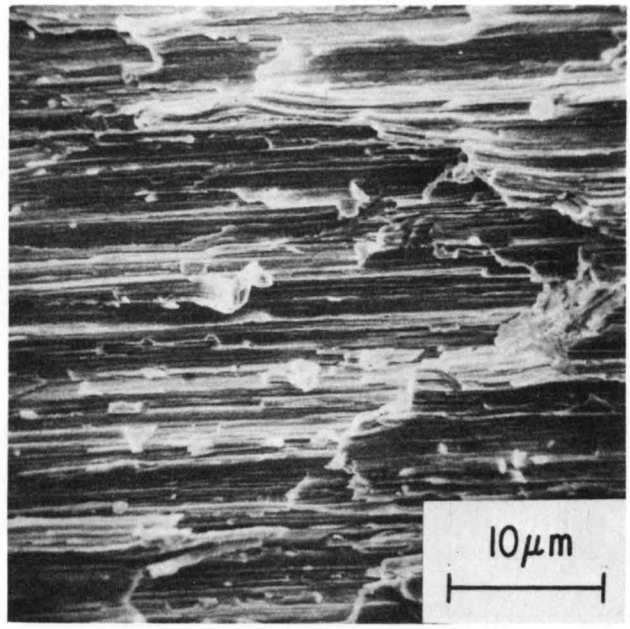


Fig. 1a SEM Fractograph of As-Drawn Doped Wire.

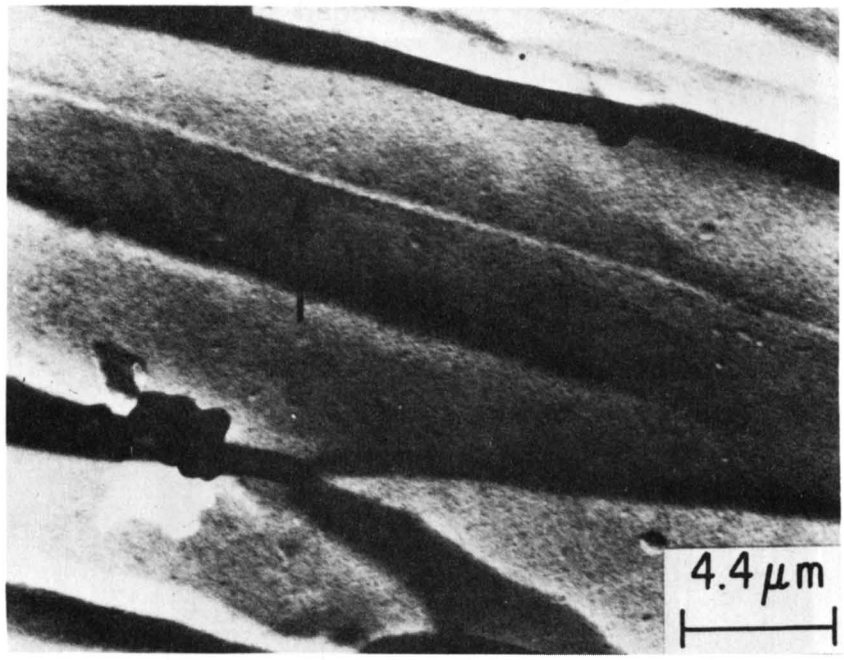


Fig. 1b TEM Replica Fractograph of Doped As-Received Wire. Arrow indicates second phase.

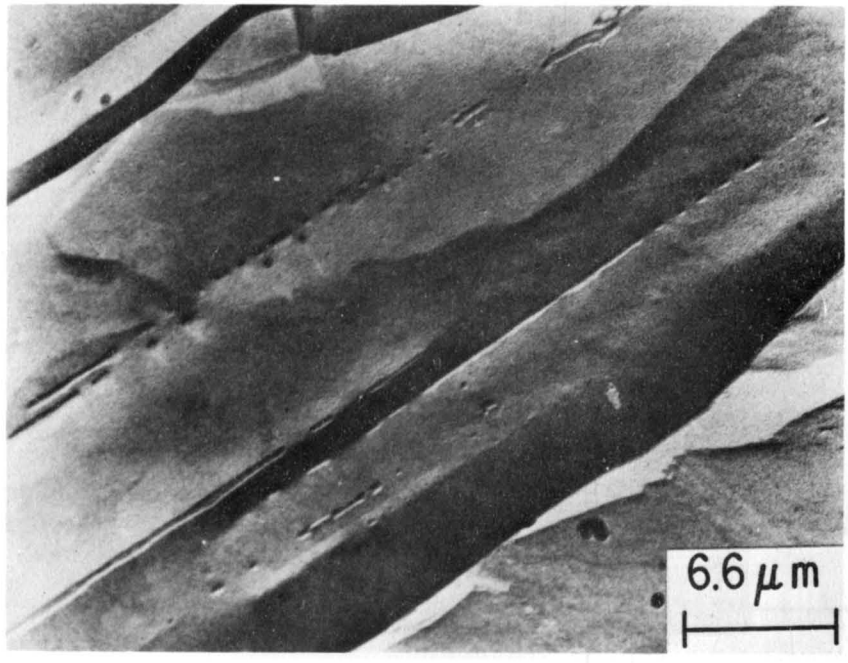


Fig. 1c TEM Replica Fractograph of Doped Wire Annealed at 800 °C.

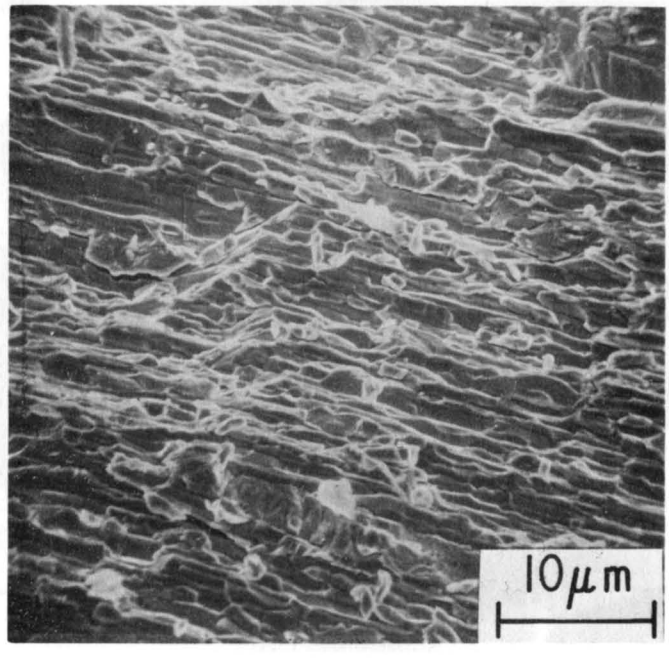


Fig. 1d SEM Fractograph of Undoped Wire Annealed at 1050 °C.

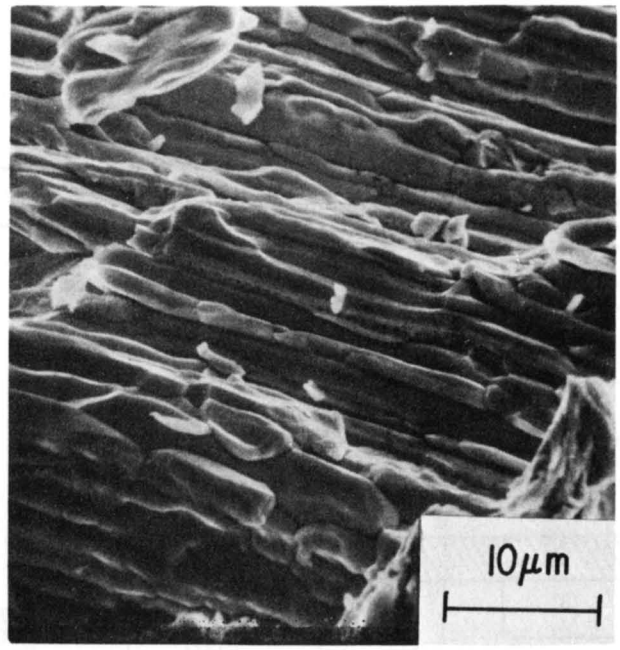


Fig. 1e SEM Fractograph of Doped Wire Annealed at 1300 °C. Arrow indicates a row of second phase bubbles.

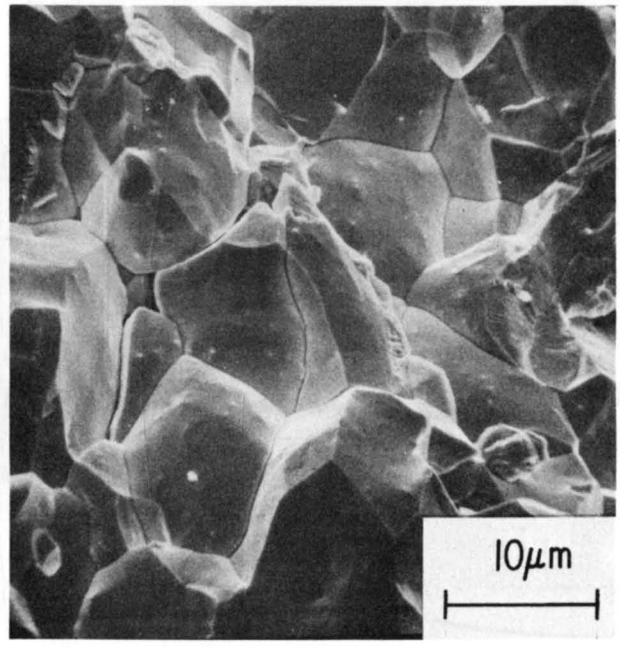


Fig. 1f SEM Fractograph of Undoped Wire Annealed at 1300 °C.

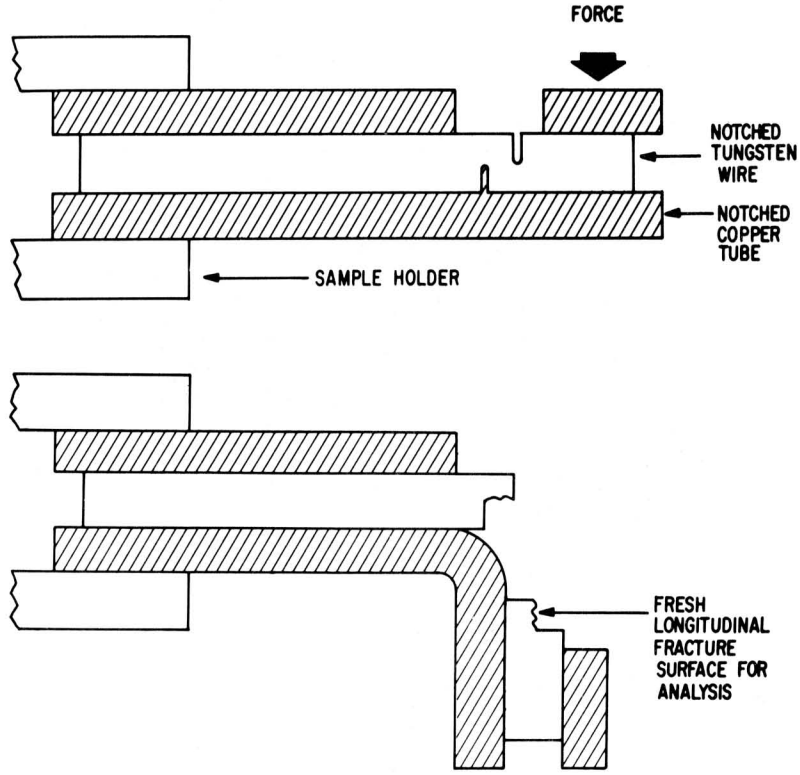


Fig 2 Longitudinal Fracture of Wire for Auger Analysis

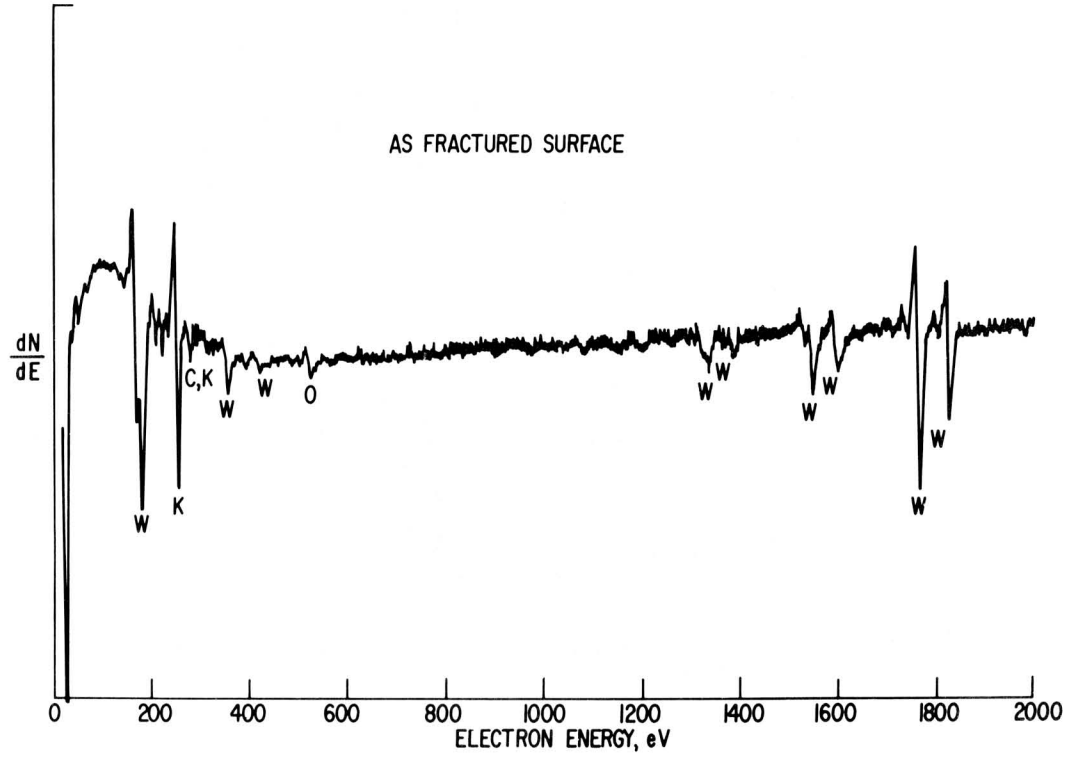


Fig. 3 Auger Spectrum of Lamp Doped Wire Annealed at 1050 °C Tungsten, potassium, oxygen, and carbon peaks identified

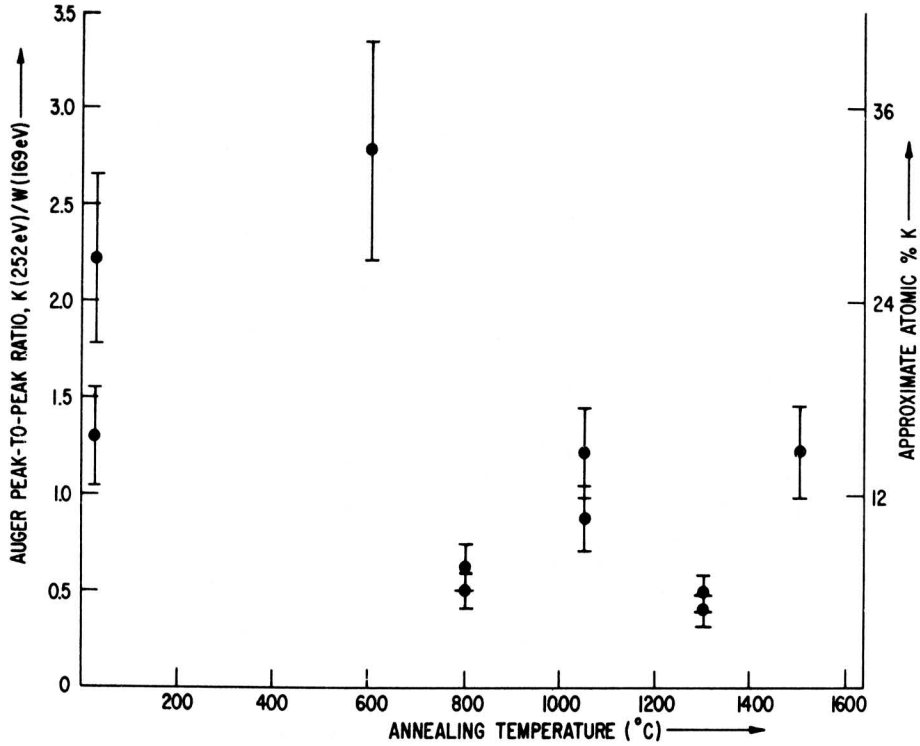


Fig. 4a Ratio of Potassium Auger Peak (252eV) to Tungsten Auger Peaks (169eV Manifold).

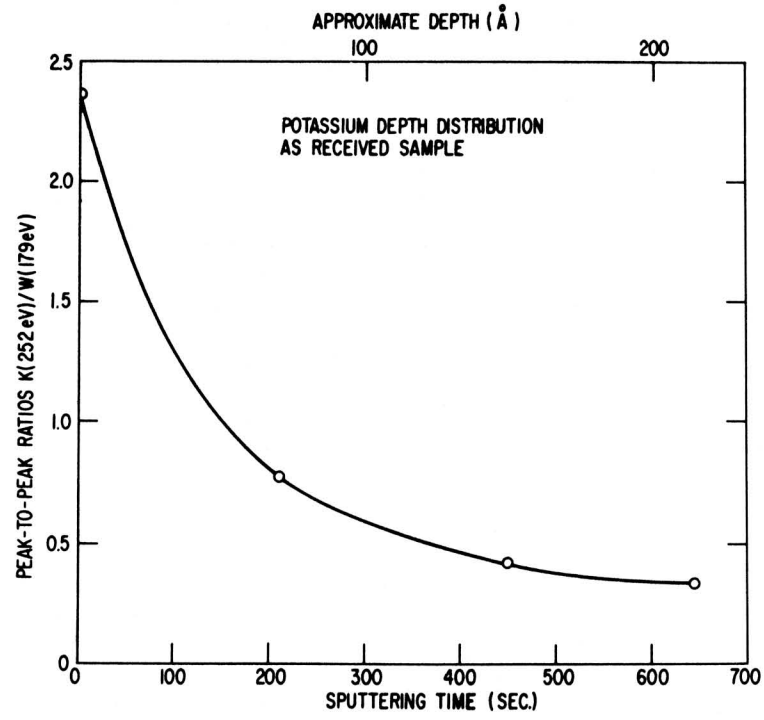


Fig. 4b Ratio of Potassium Auger Peak (252eV) to Tungsten Auger Peaks (169eV Manifold) versus Sputtering Time for As Received Doped Wire.

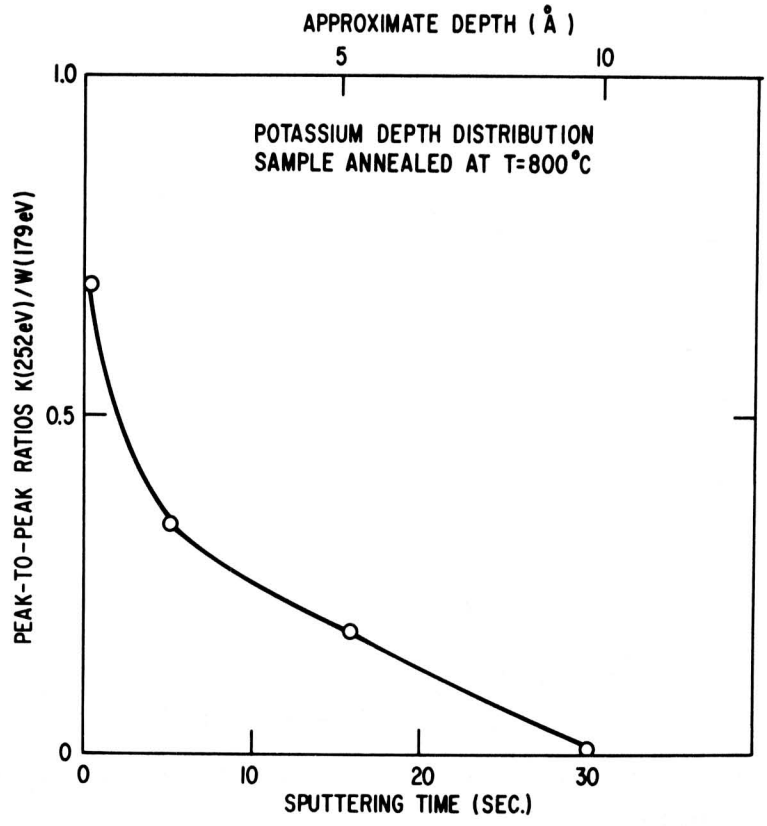


Fig. 4c Ratio of Potassium Auger Peak (252eV) to Tungsten Auger (169eV Manifold) versus Sputtering Time for Doped Wire Annealed at 800 °C. Profile is Characteristic for All Wires Annealed at 800 °C and Above.

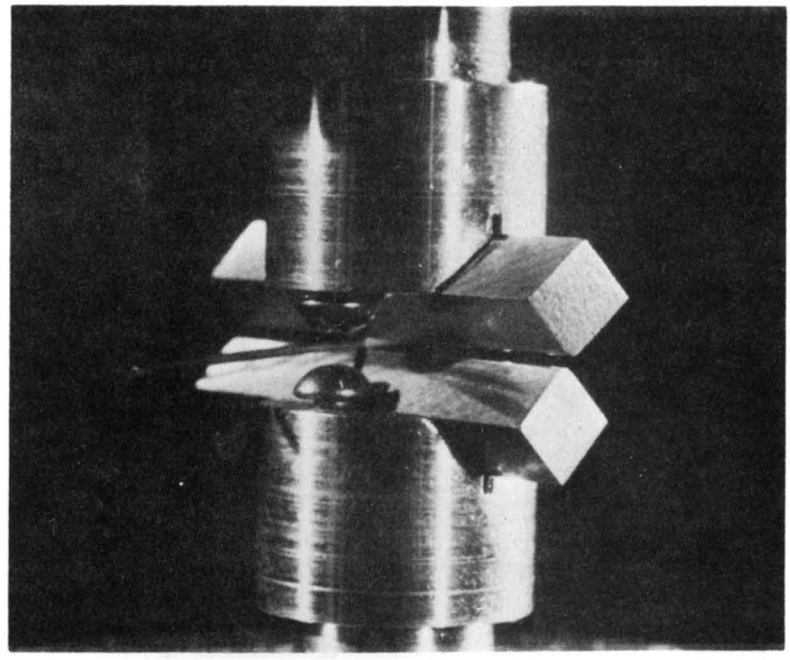


Fig. 5a Delamination Load Determination.

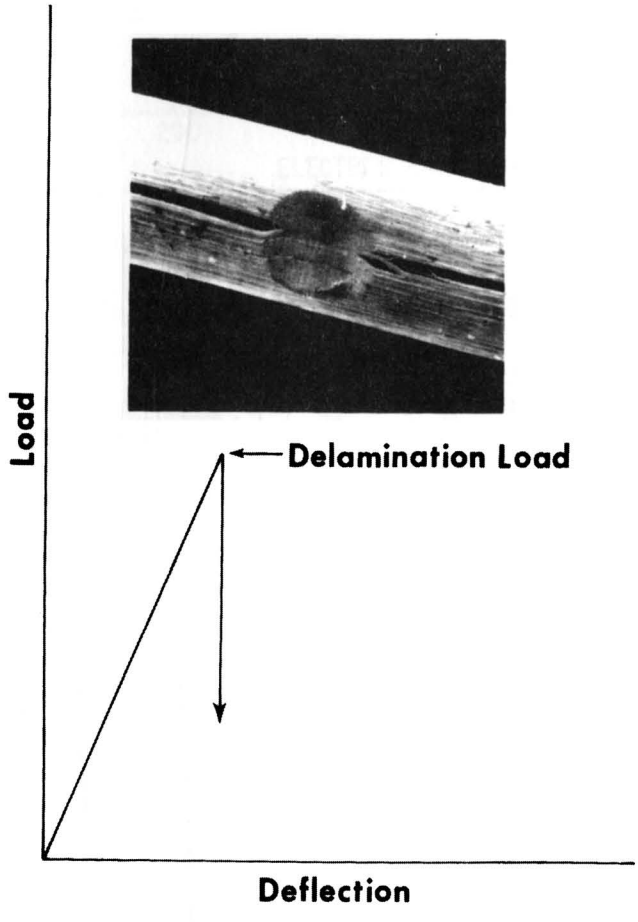


Fig. 5b Load-Deflection Record and Delaminated Wire.

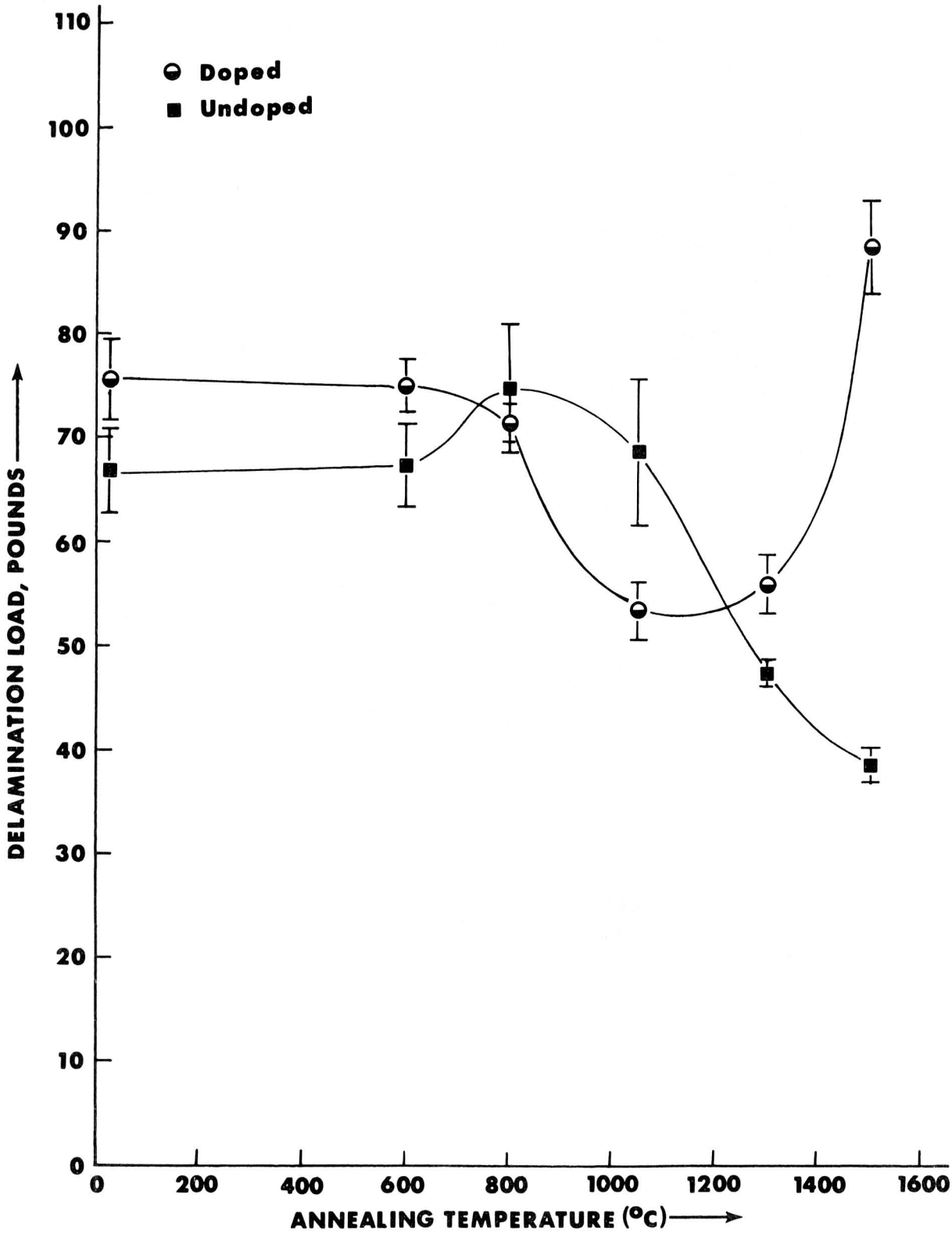
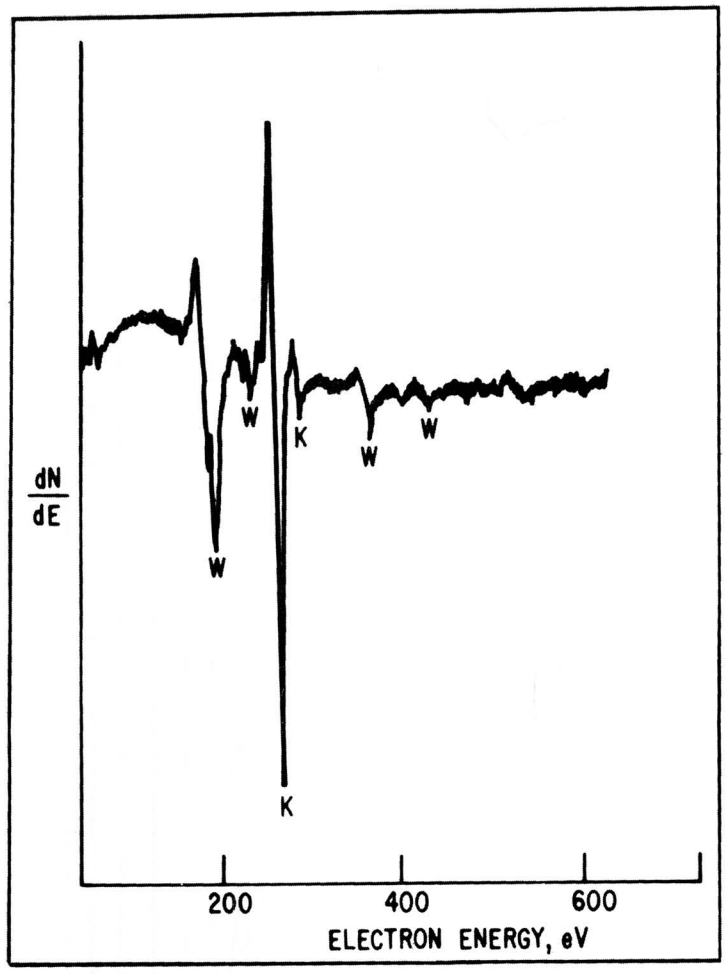


Fig. 6 Delamination Load versus Annealing Temperature



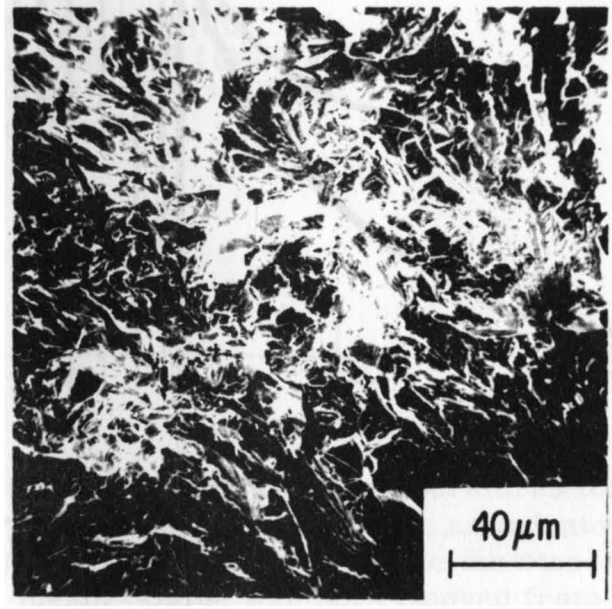


Fig. 7 Auger Spectrum and SEM Fractograph of Doped Tungsten Rod.

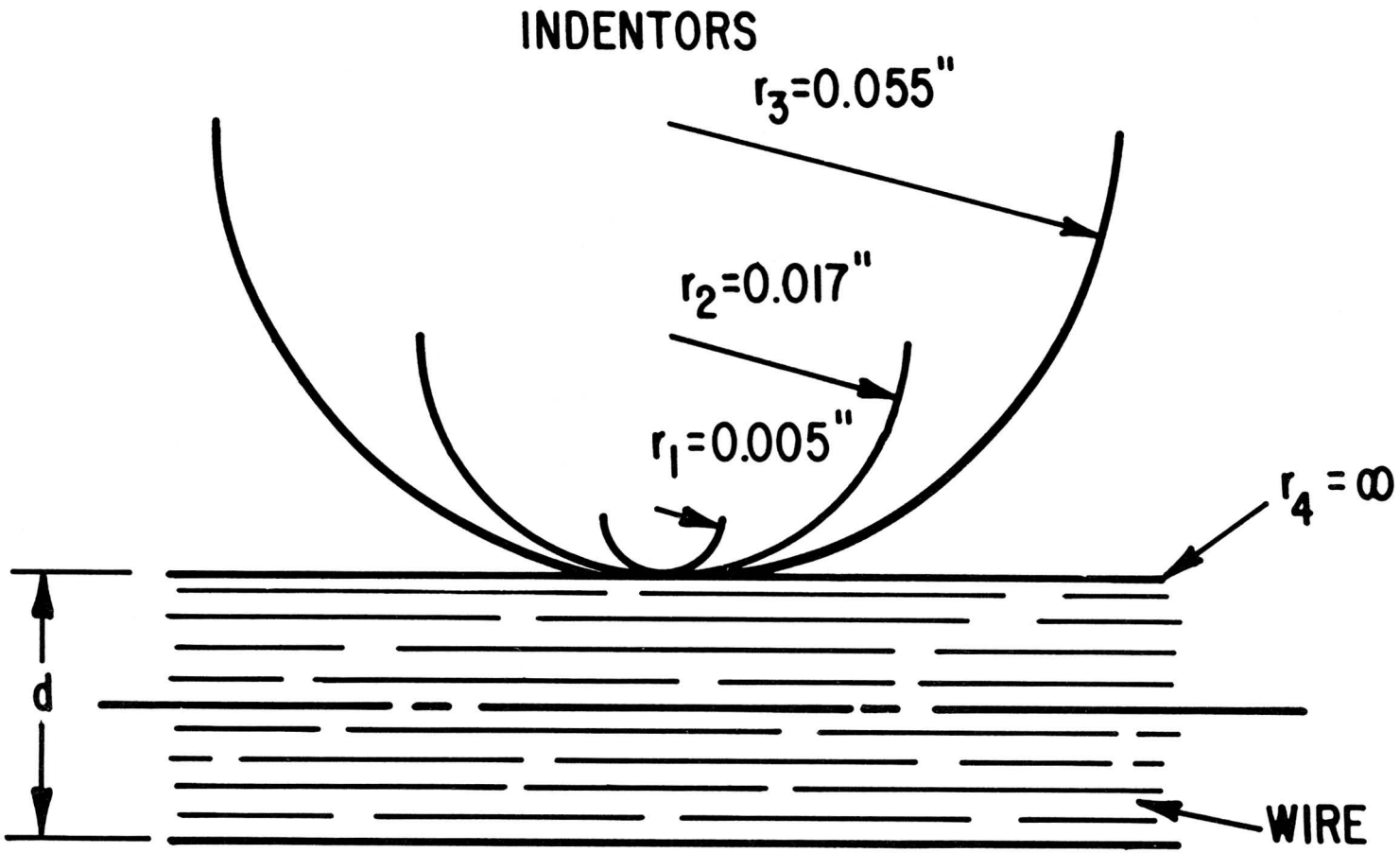


Fig. A-1 Definition of Indentor Radius, r, and Wire Diameter, d.

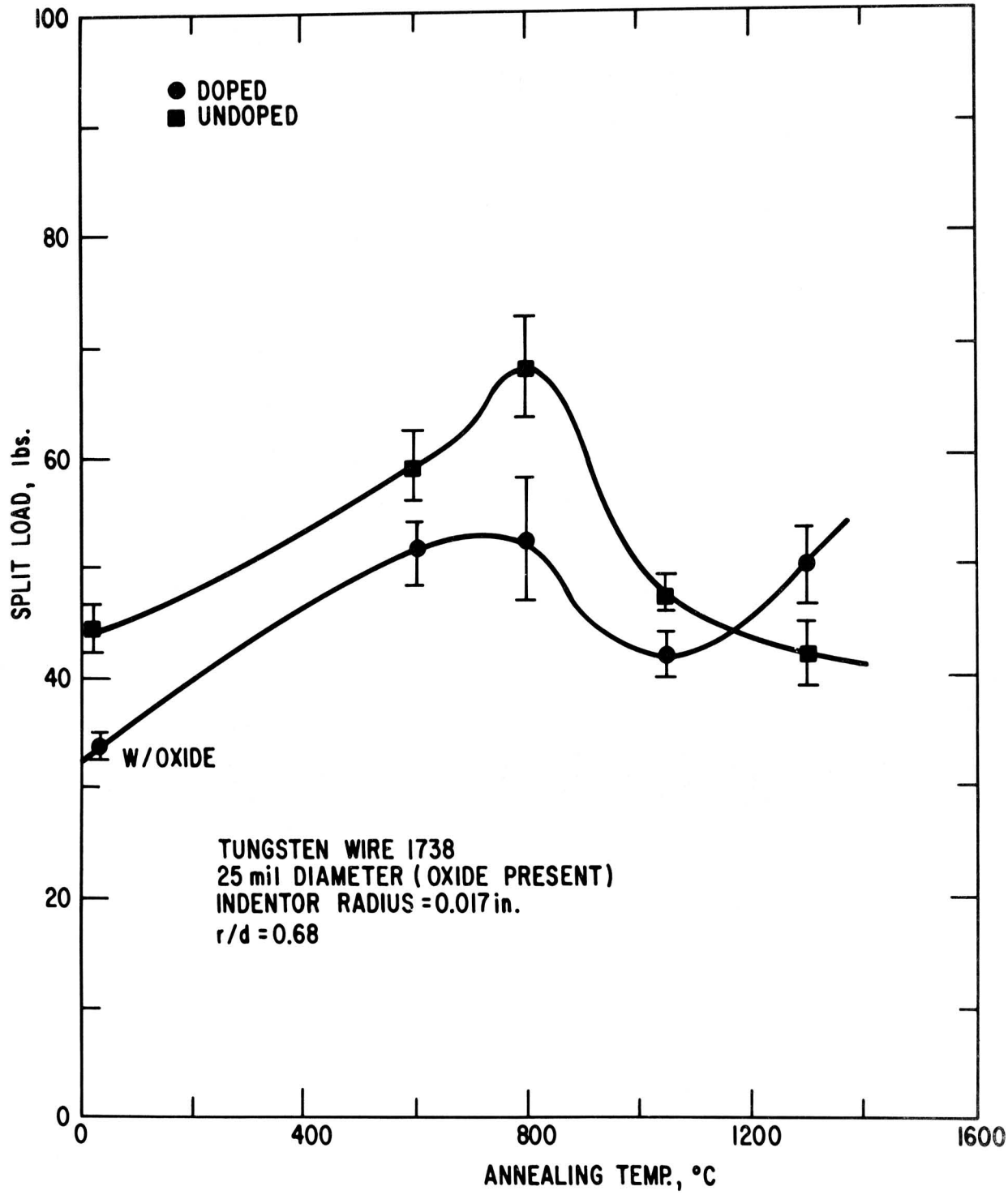


Fig. A-2 Split-load vs Annealing Temperatures for #1738 Wire (25 mil diameter) Tested with an Indentor Radius of 0.017 in. Error bars indicate the standard error of the mean Oxide was not removed from the wire in these tests.

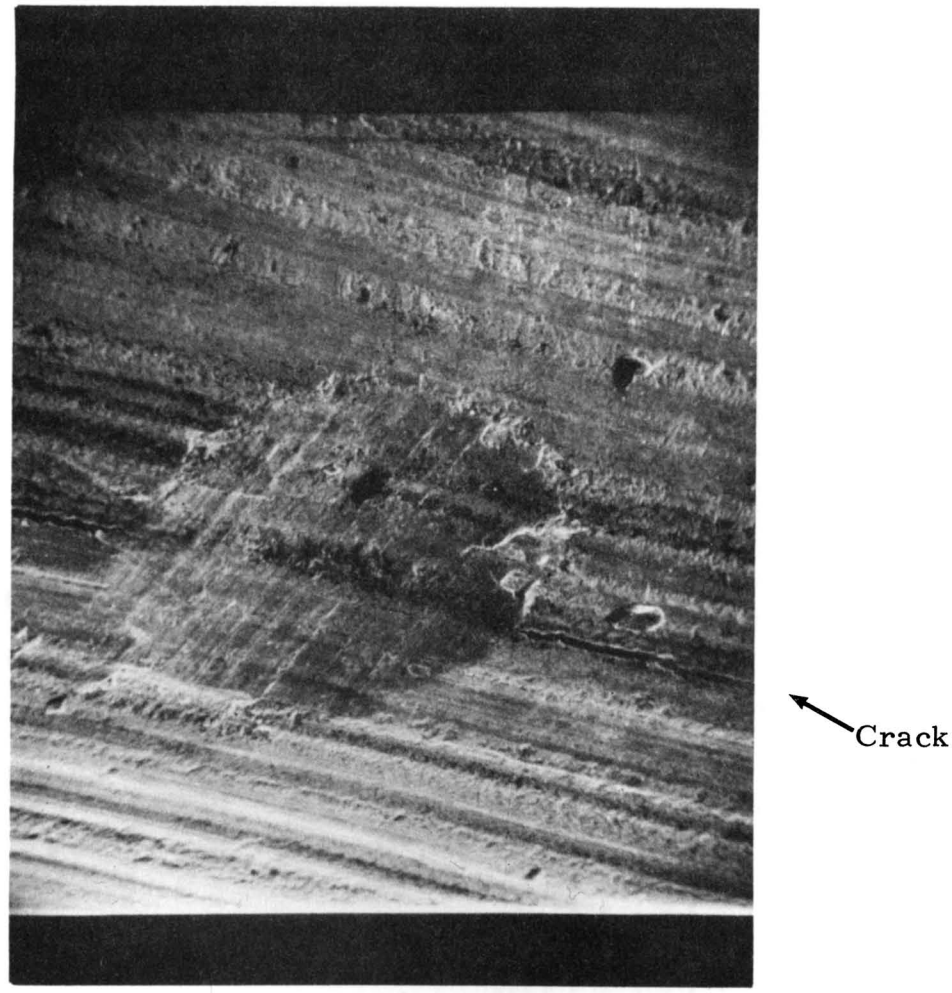


Fig. A-3 Scanning Electron Micrographs of Fractured 25 mil Doped Tungsten Wires: (a) An Early Stage of Cracking in the As-Received Condition, (b) An Early Stage and (c) Fully Developed Cracks for Wires Annealed at 1050 °C for Two Hours.

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