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STRENGTH IN A TWO-PHASE MODEL SYSTEM WITH FIBER REINFORCEMENT

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Gerd Einmahl
(M.S. Thesis)

May 2, 1966

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STRENGTH IN A TWO-PHASE MODEL SYSTEM WITH FIBER REINFORCEMENT

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May 2, 1966

ABSTRACT

Model systems consisting of a glass matrix reinforced by randomly oriented tungsten or nickel fibers with an aspect ratio of 50 were fabricated by vacuum hot-pressing and tested by bending. The systems were distinguished by interfacial bonding and different thermal expansion coefficients of the matrix and the dispersed phase.

In systems that could be densely fabricated the strength was found to increase linearly with increasing volume fraction of fibers. Reversing the sign of the thermal expansion coefficient of the matrix relative to that of the inclusions had little effect on the strength. A greater strengthening effect was achieved in the tungsten-glass systems where an interfacial bond existed as opposed to the nickel-glass series. The strengthening effect of the nickel fibers is explained by gripping of the fiber ends by the glass due to irregularities in shape from the cutting operation used to obtain short fibers.

I. INTRODUCTION

The current interest in composite materials has been in fiber glass reinforced plastics for low temperature environments and in oxide whisker reinforced metals for elevated temperatures.[1] In both cases the matrix material displays reasonable ductile mechanical behavior. In ceramic matrix composites the brittle nature of the matrix imposes entirely different criteria necessary for the formation of high strength systems. The high temperature stability of ceramics, however, makes these systems attractive.

Considering a brittle matrix containing dispersed inclusions the factors which can contribute to the mechanical strength are:

(1) The volume fraction of the dispersed phase,

(2) The size and shape of the dispersed particles,

(3) The interfacial bonding between the matrix and the dispersed phase,

(4) The difference between the thermal expansion coefficients between the phases,

(5) The elastic properties of the two phases of the composite, and

(6) Any porosity existing in the composite system.

Fabrication of composite systems has been a limiting factor in developing suitable systems in which various theories of dispersed phase strengthening can be tested. Fulrath[2] proposed a model consisting of included particles in a glass matrix fabricated by vacuum hot-pressing as a reasonable system to study the factors influencing the strength of brittle matrix composites. When alumina particles with an average size

of 2.5 microns were hot pressed into a glass of matching thermal expansion, the strength of the composite was linearly related to the volume fraction of the dispersed phase until porosity was encountered at approximately 50 v/o alumina. The bend strength of diamond sawed glass (surface damage) was found to be approximately 6500 psi, whereas a composite of 50 v/o alumina had a strength of 32,000 psi. Hasselman and Fulrath[3] postulated that particles dispersed in a glass matrix limit the possible size of Griffith's flaws in the matrix, hence raising the stress required for fracture. This mean free path will be shorter, the smaller the size of the inclusions at a constant volume fraction of dispersed phase. Jacobson[4] introduced nickel micro spheres with an average diameter of 58 microns into glasses with thermal expansion coefficients either higher or lower than that of the dispersion. A strengthening effect was found when the thermal expansion of the matrix was higher than that of nickel, and a weakening effect in the reverse case. Since no interfacial bonding existed between the nickel and the glass, the spheres shrunk away from the low-expansion matrix, thus creating porosity with its marked effect on strength. Nason[5] placed spherical tungsten particles in a glass matrix. A strong interfacial bond was developed between the glass and tungsten. In this case strengthening of the matrix was observed regardless of the mismatch of the thermal expansion coefficients. Nason's and Jacobson's results are summarized in Figs. 1 and 2. In this study the dependence of the strength of the matrix on the particle shape was studied. Fibers with an aspect ratio of 50 of nickel and tungsten were introduced into glass

matrices which, according to Jacobson and Nason should give systems with and without interfacial bonding whether formed mechanically between the matrix and dispersed phase or chemically.

Extensive work has been done on fiber reinforced matrices. It is generally recognized that parallel aligned and, if possible, continuous fibers of a high strength material greatly increase the uniaxial tensile strength of a ductile matrix.[6] Systems of this kind have been expressed mathematically[7] and an extended review on fibrous composition has been given by Sutton and Chorne.[8]

The disadvantage of the systems mentioned above is that they are strengthened only in the direction of the fiber alignment. Possible weakening normal to the fiber alignment may be observed. So far, little work has been done to study isotropic systems with fiber reinforcement. Jackson and Cratchley[9] demonstrated the angular relationship between uniaxial tensile strength and fiber orientation. Tinklepaugh et al[10,11] in studies of ceramic matrices reinforced with metal fibers oriented at random reached the conclusion that although the matrix fails at a certain stress level, the composite can still carry some load until the fibers break or are pulled out of the matrix.

II. EXPERIMENTAL PROCEDURE

A. Material Preparation

Table I shows composition, thermal expansion, and density of the materials used in this study. The glasses were smelted from their oxides or carbonates in an inclined, rotating platinum crucible. The quenched material was ground down to a particle size below 10 microns. The glasses were chosen on the basis of their thermal expansion relative to that of tungsten and nickel. The thermal expansion coefficient of the glasses was measured by means of a silica-rod dilatometer. The density of the glasses was measured after having vacuum hot-pressed them at 2000 psi slightly above the softening point.

Tungsten and nickel were obtained from the Sylvania Electric Company and the C. O. Jelliff Manufacturing Corporation respectively. The material was supplied as high purity wire 0.002 inches in diameter, rolled in lengths of several miles. To obtain fibers of a length of 0.1 inch, the wire was rewound onto a wooden drum, and each layer of windings was coated with Duratite household cement diluted with acetone. The windings were then cut with a thin abrasive blade. After dissolving the cement in acetone the fibers were ready for use. This procedure was selected in order to obtain the large quantities of fibers needed and to secure the fiber ends as square as possible. In spite of the extreme care taken in cutting, some plastic flow, particularly in the case of nickel, could not be avoided completely (reference to this point will be made in discussing the results).

B. Sample Fabrication

Four test series were fabricated: tungsten fibers dispersed in glass A and glass B in amounts of 5, 10, 15, 20, and 25 v/o, and similarly nickel fibers dispersed in glass B and glass C in quantities of 5, 10, 20, 30, 40, and 50 v/o. The maximum volume fraction of dispersed fibers in these series was limited by the compactability of random fibers. Jackson and Cratchley[9] state that complete randomization of the fiber orientation leads to a packing density not higher than 20 - 30 v/o of the fibrous phase. The exact value depends on the aspect ratio of the fibers and on the stiffness of the material. Cold pressing at 3000 psi of the nickel and tungsten fibers as specified in the foregoing paragraph showed a compactability of 50 and 25 v/o of theoretical density for the nickel and tungsten fibers respectively.

The appropriate amounts of fibers and dried glass powder were dry mixed in a jar rotated around its diagonal axis. From this a disk 2 inches in diameter and about 0.07 inch thick was hot pressed in vacuo in a graphite die. The hot-pressing conditions were adjusted to obtain a specimen with a bulk density as close as possible to the calculated theoretical density of metal fiber and glass. The applied pressure varied from 1500 to 3000 psi and was higher for the samples with tungsten fibers than for the ones with an equal nickel content. The hot-pressing temperature was determined from the softening point of the glasses and from the temperature at which devitrification occurred as detected by X-ray diffractometry. The formation of voids due to an elastic relaxation of the fiber compact was avoided by cooling the specimen under the applied load down to about 400° C. This step was not

possible for samples with fiber loads of 5 and 10 v/o because the system was not sufficiently strengthened to avoid cracking on cooling under pressure. In this case a density of 100% of the theoretical density was easily achieved without cooling under applied pressure.

The samples were surface ground with 400 and then 600 grit SiC grit in order to remove the adherent graphite layer and give a uniform surface damage. From the disks, bars with a width of 1/4 inch were cut with a diamond cut off wheel running at a speed of 2800 surface feet/minute. Due to the high ductility of nickel, a smearing effect of the fiber ends occurred on the cut faces of the samples, and was eliminated by grinding these faces with 400 and 600 grit corundum paper. In the light of the work by Nason[5], in which he showed that different surface treatments of samples similar to the ones used here did not change the strength significantly, it was felt that an annealing of the samples after cutting was not necessary.

C. Sample Testing

The modulus of rupture was determined by measuring the load under which the sample failed in a four-point bending device and calculating the modulus of rupture from:

$$\sigma = Mc/I$$

where

σ = Outer fiber stress or modulus of rupture,

M = Bending moment,

c = Distance from the surface of the sample to the neutral fiber,

I = Moment of inertia of the bar cross section about the neutral axis.

The load was applied with a cross head speed of .04 inch/min. The load bearing surface of the sample had been parallel to the axis of load application during the hot-pressing. A load vs. deflection curve was automatically recorded. Typical curves are shown in Figs. 6, 7 and 9. In order to bring about the characteristical features of the fibrous dispersion, a fairly low magnification using a metallurgical microscope was employed (see Fig. 5). No special surface treatment of the samples was necessary.

The density was determined by measuring the bulk density and relating it to the theoretical value calculated from the density of the hot pressed glass and that of the metal as indicated by the manufacturer. Since the samples had a well defined geometrical shape, the bulk density could be determined by measuring the dimensions of the sample and its weight.

The Young's modulus was found by means of the resonance technique as described by Spinner and Thefft.[12] From the frequencies obtained and the dimensions of the sample, Young's modulus was calculated using appropriate tables.[13] The dimensions of the sample, limited by the size that could be hot pressed did not allow the shear modulus and hence the Poisson's ratio to be determined.

III. EXPERIMENTAL RESULTS

The results of the strength measurements of the systems with tungsten and nickel fibers are listed in Tables II and III respectively. Two values are shown for the strength of the samples, one for the maximum strength observed and one determined when the load-deflection curve departed from linearity. These values are described as the modulus of rupture and modulus of matrix failure respectively and are plotted in Figs. 3 and 4. The slopes have been drawn into the figures as calculated by a least square fit. Each data point is the mean value of 10 to 25 tests. It was found that there is negligible difference in the slope of the modulus of rupture and modulus of matrix failure for the two tungsten series, whereas for the nickel series the ratio of the slope of the modulus of rupture and that of matrix failure is 1.69 for glass B and 1.53 for glass C. It was not possible to fabricate completely dense samples with higher volume fractions of dispersions as shown in Tables II and III. Therefore, the very large standard deviations observed were a result of the increased porosity and resultant affect of porosity on strength. The data points that did not fit the linearity were not used in calculating the slope.

The strength values show that a minimum amount of fiber dispersion is needed in order to strengthen the system. This is in accord with Cratchley's[14] theoretical plot of the ultimate tensile strength vs. volume fraction of fibers in a metal matrix reinforced by refractory metal fibers. There the amount of dispersion at which the system has its lowest strength was found to be 5 v/o. In the present experimental work, no attempt has been made to find out what amount between 0 and

10 v/o fiber load is the critical value for the lowest strength.

There appears to be no simple explanation for the strengthening behavior in terms of the thermal expansion coefficients of the phases relative to each other. In the case of the tungsten series with the glass A whose thermal expansion is less than that of the inclusions greater strengthening is observed than in the glass B system with a higher thermal expansion. The opposite effect was achieved in the nickel series.

The mode of fracture is different in the two metallic fiber series as shown in Fig. 8. In the nickel-glass system the fracture propagates perpendicular to the neutral axis, whereas the tungsten-glass system shows a delaminated fracture. This pattern was observed in all specimens. Fig. 9 represents the load vs. deflection curves of the two bars shown in Fig. 8.

From Fig. 5 showing typical microstructures of tungsten and nickel samples with 20 v/o of dispersion it can be seen that there is not complete randomness of fiber orientation. Figure 10 shows that the randomness was two dimensional by comparison of the strength of samples cut so that the tensile surface was either the hot pressed face or a plane perpendicular to it. In no case was the tensile stress applied perpendicular to the two dimensional fiber array.

The shape of the load vs. deflection curves, typical examples of which are presented in Fig. 6 for the nickel-glass B series and in Fig. 7 for the tungsten-glass B series, appears to depend upon the kind of metal used as a dispersion. The samples with 5 and 10 v/o nickel even exhibited stress-strain curves similar to a material with an upper

and lower yield strength. The strain between the failure of the matrix and the point of the highest strength is greater for nickel samples than for the ones with tungsten fibers. Many nickel reinforced glass samples failed to completely separate on bending due to the plastic deformation of nickel fibers across the fracture.

The results of the measurement of Young's modulus for the tungsten-glass A system are listed in Table IV. Figure 11 is a plot of the elastic modulus vs. volume fraction of tungsten fibers. At low volume fractions of fibers a linear relationship slightly beneath the upper bound of elasticity as calculated by Paul[15] is observed. The points for 15 and 20 v/o dispersion fall below that line because of the increase in porosity as shown in the figure.

Elastic modulus determinations by the sonic method could not be made on nickel reinforced glass samples due to erratic resonant frequencies and difficulties in diamond sawing suitable samples.

IV. DISCUSSION

The results in Figs. 3 and 4 indicate that the presence of a strong interfacial bond between the matrix and the inclusions, as it occurs in the tungsten-glass systems, contribute significantly to the strength of the composite. It is generally recognized that the strengthening effect of fibers reinforcing a matrix lies in the fact that stress is transferred to the fibers through the matrix. This presumes that the fibers are stiffer than the matrix. The better the interfacial bond between the matrix and the dispersed phase the more stress is transferred. The fibers can carry more stress the higher their elasticity is relative to that of the matrix. For the tungsten-glass systems investigated Young's modulus of the fibers is 4 and 7 times as high as that of the glass and for the nickel-glass series 2 and 3 times. The fraction of the total force on the system that can be carried by the fibers is a function of their volume fraction. This explains the increase of stress required for the matrix failure with increasing volume fraction of the fibers.

When during the bending test of a tungsten-glass sample the strength of the matrix is exceeded, a crack develops at the surface under tension and propagates through the glass, leaving a delaminating fracture path, as shown in Fig. 8.

The nickel-glass systems lacked such a chemical bonding at the interface. In the nickel-glass B system, the thermal expansion coefficient of the nickel was higher than that of the glass. Therefore, during the cooling after the hot pressing, the fiber shrunk away from the matrix, so that the system was incapable of transferring some

stress over the length of the fiber. It has already been mentioned that the preparation of the fibers caused some plastic flow leaving the ends of the fibers with an irregular shape, and an undefined surface roughness. These ends are capable of anchoring the fiber in the matrix, so that as an idealization the fiber may look like a dumbbell embedded in glass. In this structure some stress can also be transferred to the fibers, which explains the increase of strength with increasing volume fraction of nickel as opposed to Jacobson's results with the system nickel spheres - D glass shown in Fig. 2.

After the stress has reached the level at which the glass breaks, a plastic deformation of the fiber takes place and either it breaks apart or one of its ends slips out of the grip. This establishes the ultimate strength of the system. The fact that the sample now does not fail at once is due to the pulling of the fibers out of the matrix.

The same mechanism applies for the nickel-glass C system. The higher thermal expansion coefficient of the glass relative to that of the nickel secures a somewhat tighter gripping of the fiber, as can be seen from the slightly higher strength of this system vs. that with glass B.

The fabrication of dense samples was impossible where higher volume fractions of fibers were introduced. In these cases the composite may be considered to be a three-phase system consisting of a continuous glass matrix, a fibrous dispersed phase that enhances the strength, and pores that exert a weakening effect on the system. This is evident in Figs. 3 and 4 where the strength values for the higher

fiber fractions fall markedly below the line indicating the trend of a linear strengthening effect.

The same feature is shown in Fig. 11, where the porous phase also affects the modulus of elasticity of the system. It is interesting that the slope suggested for the Young's moduli of the samples with a low fiber content is fairly close to the upper bound proposed by Paul[15] and Hashin and Rosen[16] for a parallel alignment of fibers. This may probably be ascribed to the incomplete two-dimensional randomness of the samples investigated.

V. SUMMARY

In this study, model systems for a brittle matrix reinforced by randomly dispersed, crystalline fibers have been investigated to study the effects of internal stresses, strong interfacial bonds, and volume fraction of the dispersed phase on the mechanical strength of the composite. The parameters were the modulus of rupture as the criterion for ultimate tensile stress a fiber reinforced system can bear, and the modulus of matrix failure at which all further load has to be carried by the fibers.

Generally, both moduli were found to increase linearly with increasing fiber content, where nearly a complete density of the samples could be achieved. As in similar systems with spherical inclusions, the tungsten-glass composites having strong bonds between matrix and dispersed phase enhance strength more than the nonbonding nickel-glass systems. In spite of the random orientation of the fibrous inclusions, a theory that is accepted to cause strengthening of a matrix in which parallel aligned fibers uniaxial to the tensile stress are embedded is believed to contribute significantly to the improvement of mechanical properties of the systems studied. The strength of a fiber reinforced composite depends on the stress that is transferred through the matrix to the fibers. The amount of stress transfer can be increased by securing a strong bond between the matrix and the inclusions.

In the nickel-glass systems, the controlling mechanism of carrying load beyond the matrix failure is the strength of the fibers that are still gripped in both parts of the broken matrix. When they are

broken and pulled out of the matrix, the sample fails completely.

Because of the complexity of strains and stresses in systems as have been treated here, only a qualitative explanation is possible, and it appears that only further experimental work will discover the totality of mechanisms enhancing mechanical strength in a system consisting of a brittle matrix and randomly dispersed fibers.

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TABLE I

PROPERTIES OF MATERIALS

Material	Composition wt. %	Thermal Expansion $\times 10^{-6}$	Glass Hot Press Density g/cm^3	Metal Density g/cm^3
Glass A	67.0 SiO_2 4.5 Na_2O 28.4 B_2O_3	3.33 (R.T.-450° C)	2.16	
Tungsten		5.6 (R.T.-700° C)		19.17
Glass B	70 SiO_2 16 Na_2O 14 B_2O_3	7.7 (R.T.-450° C)	2.47	
Nickel		13.9 (R.T.-300° C)		8.9
Glass C	50 SiO_2 37 Na_2O 13 Al_2O_3	19.03 (R.T.-450° C)	2.51	

TABLE II

Moduli of rupture and matrix failure for the series tungsten fibers - glass A and tungsten fibers - glass B

v/o fibers	0	5	10	15	20	25
Glass A						
Rel. density %	100	100	100	99	96	91
Modulus of rupture psi x 10^{-3}	8.3	9.5	19	22.8	20.3	18.3
Std. deviation %	16	21	20	15.7	13.7	36.7
No. of tests	32	10	10	10	18	10
Modulus of matrix failure psi x 10^{-3}	8.3	8.2	12.2	20.9	17.8	17.9
Std. deviation %	16	18	21.6	20.3	19.2	31.5
Glass B						
Rel. density %	100	98.6	100	96	92	90
Modulus of rupture psi x 10^{-3}	9.7	5.8	9.9	12	14.7	8.3
Std. deviation %	10	13.8	12.1	9.2	13.6	29.4
No. of tests	20	10	10	16	18	10
Modulus of matrix failure psi 10^{-3}	9.7	5	8.9	10.1	13.9	7.5
Std. deviation %	10	12	14.6	14.8	15.3	28.5

TABLE III

Moduli of rupture and matrix failure for the series nickel fibers - glass B and nickel fibers - glass C

v/o fibers	0	5	10	20	30	40	50
Glass B							
Rel. density %	100	100	99	98.3	96	93	90
Modulus of rupture psi x 10^{-3}	9.7	5.9	6.8	9.5	10.5	13.3	16.3
Std. deviation %	10	9	11.8	11.3	15	14.8	17.7
No. of tests	10	10	29	16	39	24	16
Modulus of matrix failure psi x 10^{-3}	9.7	5.9	6.4	7.2	8.3	11.1	11.6
Std. deviation %	10	9	14.7	8.6	13.9	15.5	18.5
Glass C							
Rel. density %	100	100	99.3	96	94.5	93.5	80
Modulus of rupture psi x 10^{-3}	10.3	5.9	6.8	9.3	12.6	14.8	14.9
Std. deviation %	21	4.6	10.6	11.1	20.6	21.1	9.9
No. of tests	10	10	16	15	26	22	10
Modulus of matrix failure psi	10.3	5.9	6.3	7.9	9.9	11.7	11.5
Std. deviation %	21	4.6	13	9.1	19.9	23.6	12.9

TABLE IV

Elastic Properties

Glass	Inclusion Type	Inclusion Vol %	Rel. Density %	Young's modulus kbar
N-4			100	499
"	W	5	100	603
"	"	10	100	710
"	"	15	97	748
"	"	20	94	790

FIGURE CAPTIONS

Fig. 1 Modulus of rupture for the systems tungsten-glass N-2 and tungsten-glass N-4 vs. volume fraction of tungsten spheres of a diameter 25 to 38 u (after Nason)[5].

Fig. 2 Modulus of rupture for the systems nickel-glass 8 and nickel-glass D vs. volume fraction of nickel spheres with average diameter of 48 u (after Jacobson)[4].

Fig. 3 Outer fiber stress for the systems glass C-nickel and glass B-nickel vs. volume fraction of nickel fibers.

Fig. 4 Outer fiber stress for the systems - glass B-tungsten and glass A-tungsten vs. volume fraction of tungsten fibers.

Fig. 5 Microstructures of samples with 20 v/o fiber reinforcement.

A: With nickel fibers as viewed in direction of hot pressing.

B: With tungsten fibers as viewed in direction of hot pressing.

C: With tungsten fibers as viewed perpendicular to the hot pressing direction.

Fig. 6 Typical load deflection curves of the nickel-fiber reinforced samples. The numbers on the curves indicate the volume fraction of dispersion.

Fig. 7 Typical load deflection curves of tungsten-fiber reinforced samples. The numbers on the curves indicate the volume fraction of dispersion.

Fig. 8 Typical form of crack appearance for samples with tungsten fibers (upper sample) and nickel fibers, as shown on the examples of 20% nickel in glass B and 20% tungsten in glass A.

Fig. 9 Load deflection curves for the samples pictured in Fig. 8.

Fig. 10 Modulus of rupture of the system - blass B-nickel vs. volume fraction of nickel fibers with different loading faces relative to the hot-press face.

Fig. 11 Young's modulus and relative density of the system tungsten-glass A vs. volume fraction of tungsten fibers. The crosses represent theoretical modulus calculated after Paul.

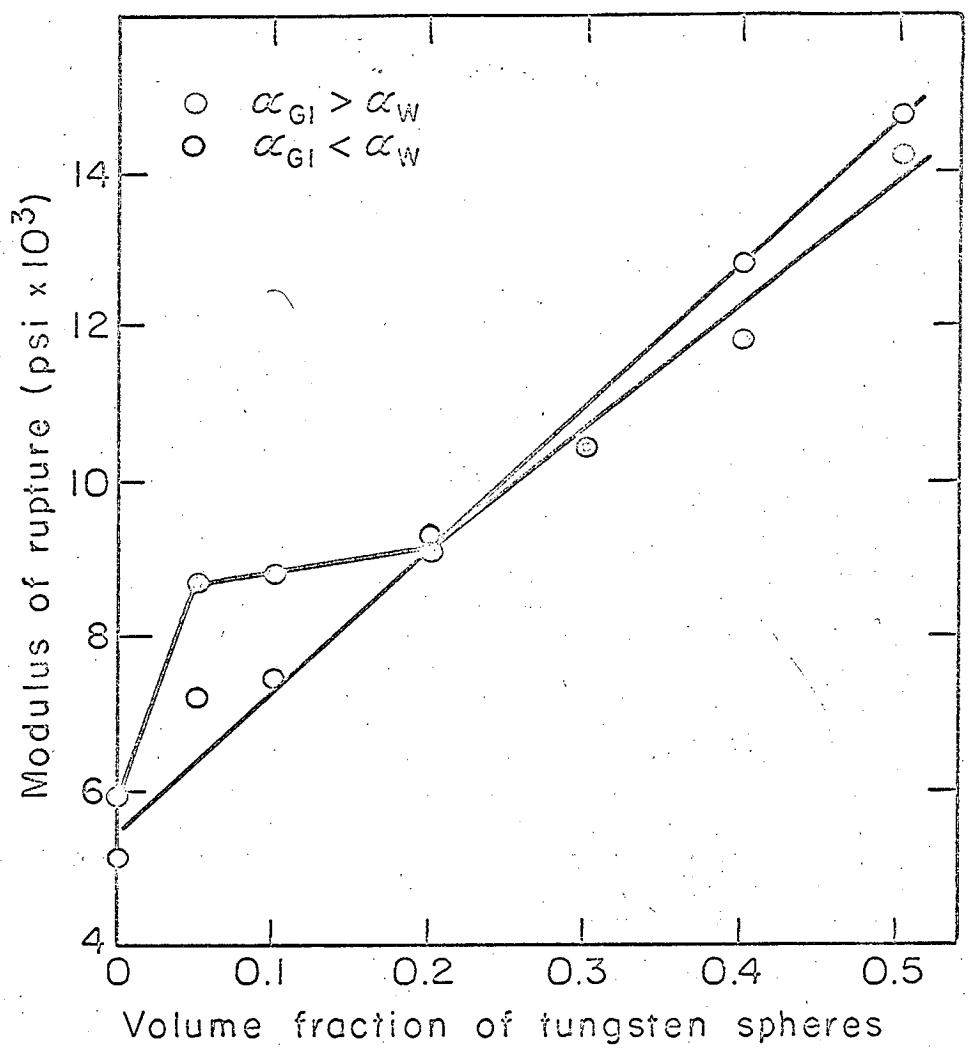

FIGURE I.

MUB-10432

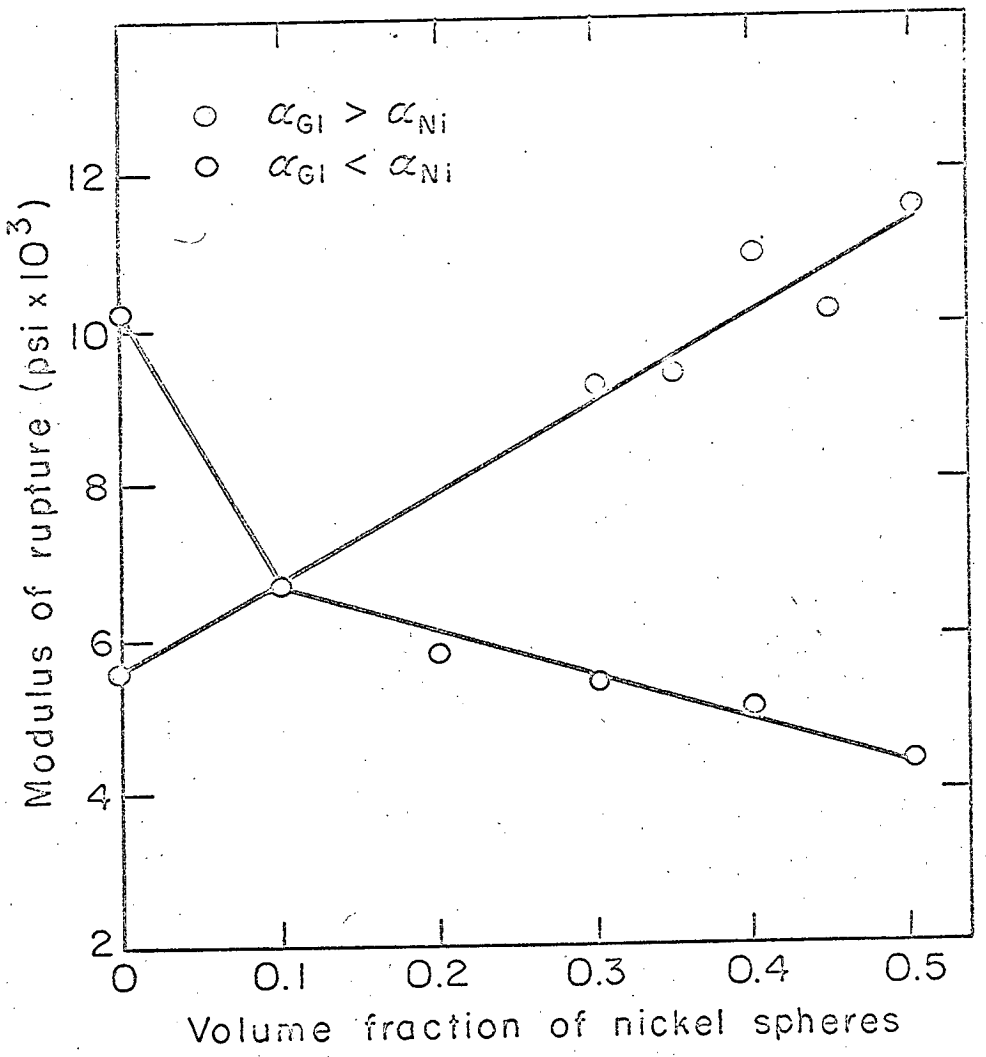

FIGURE 2.

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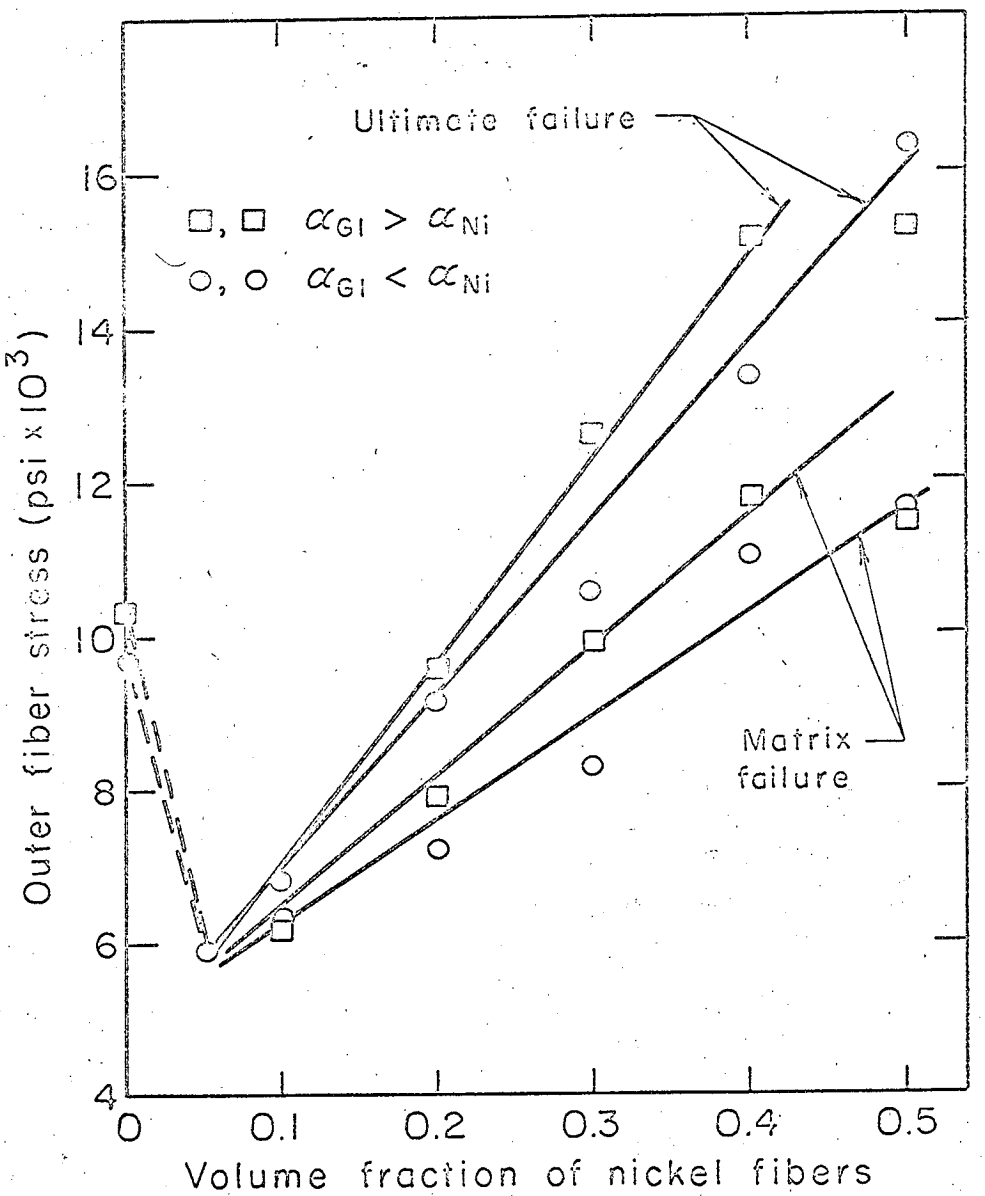

FIGURE 3.

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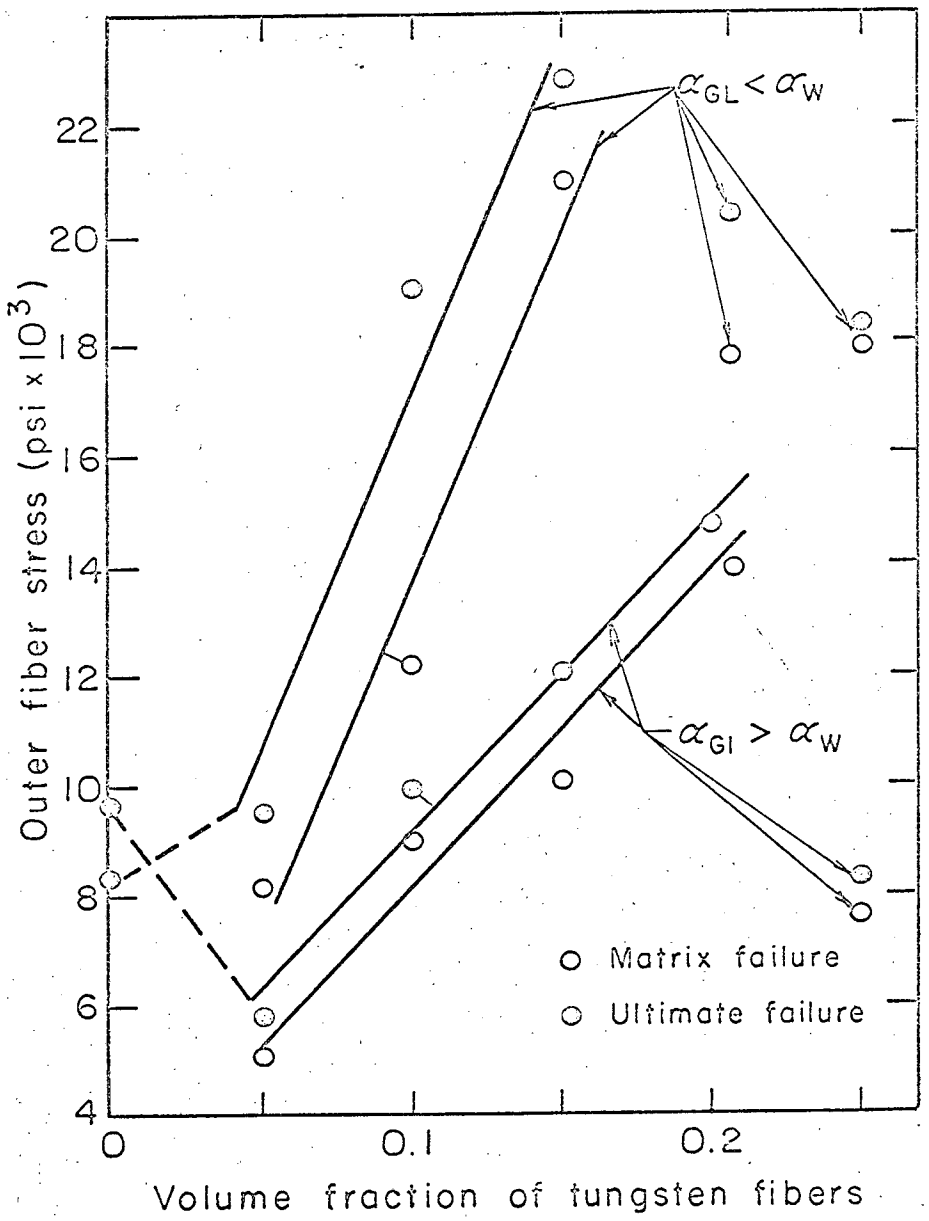

FIGURE 4.

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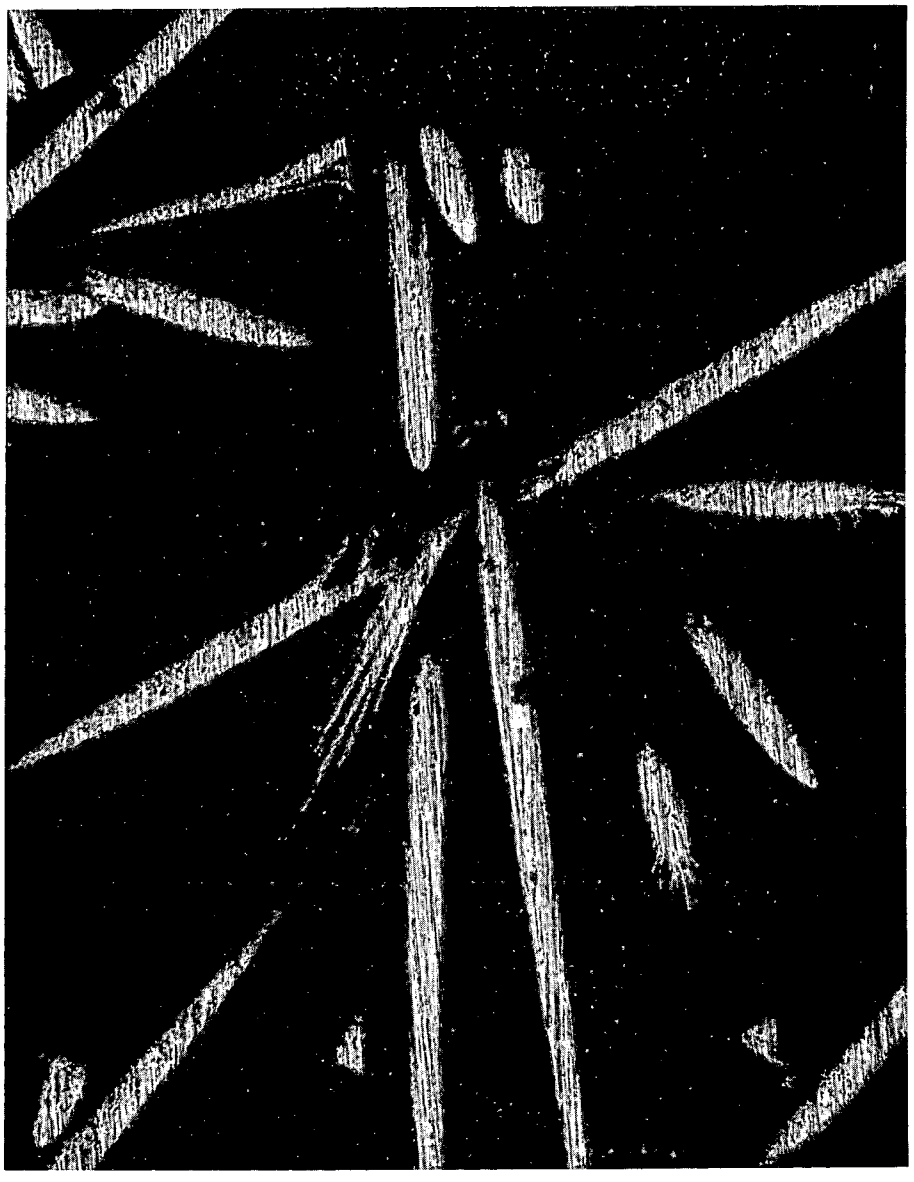
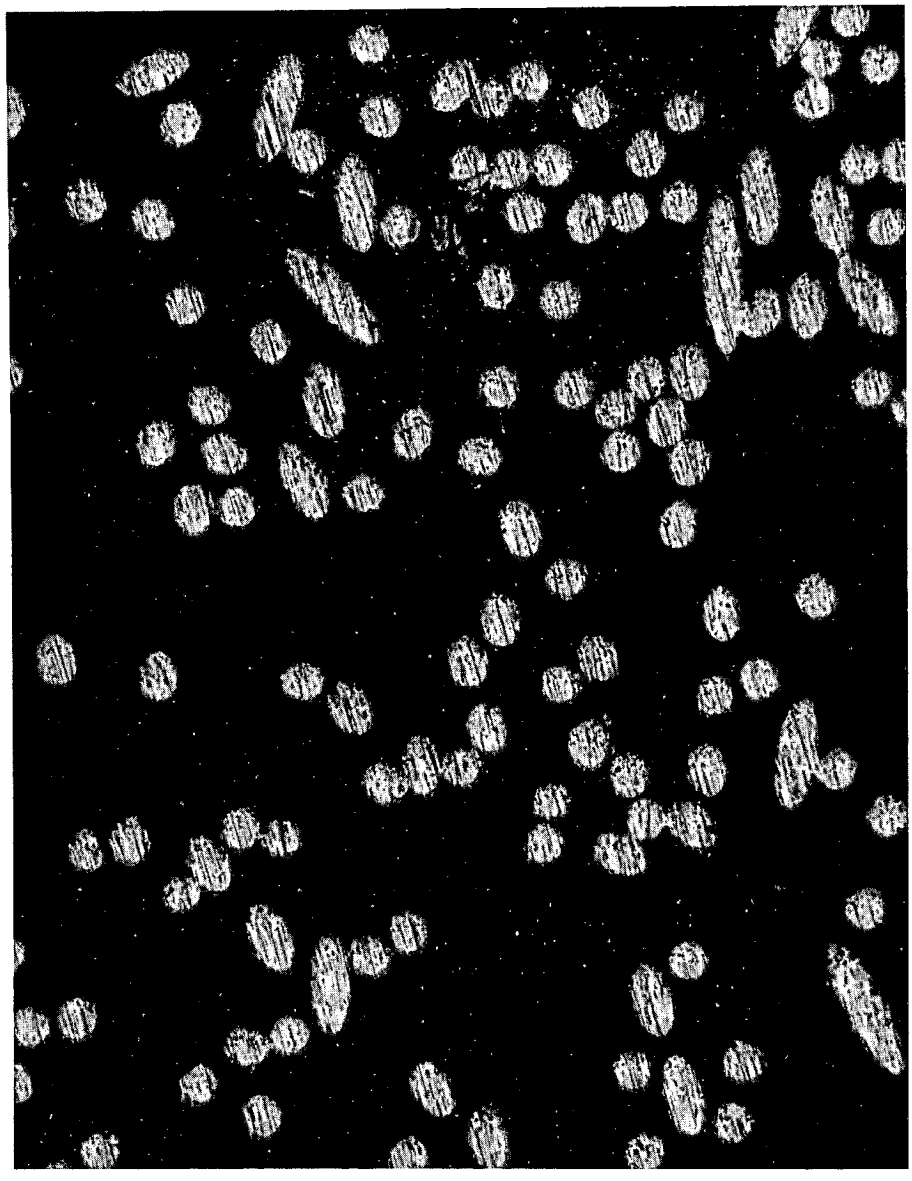
ZN-5567

Fig. 5a



ZN-5568

Fig. 5b



ZN-5569

Fig. 5c

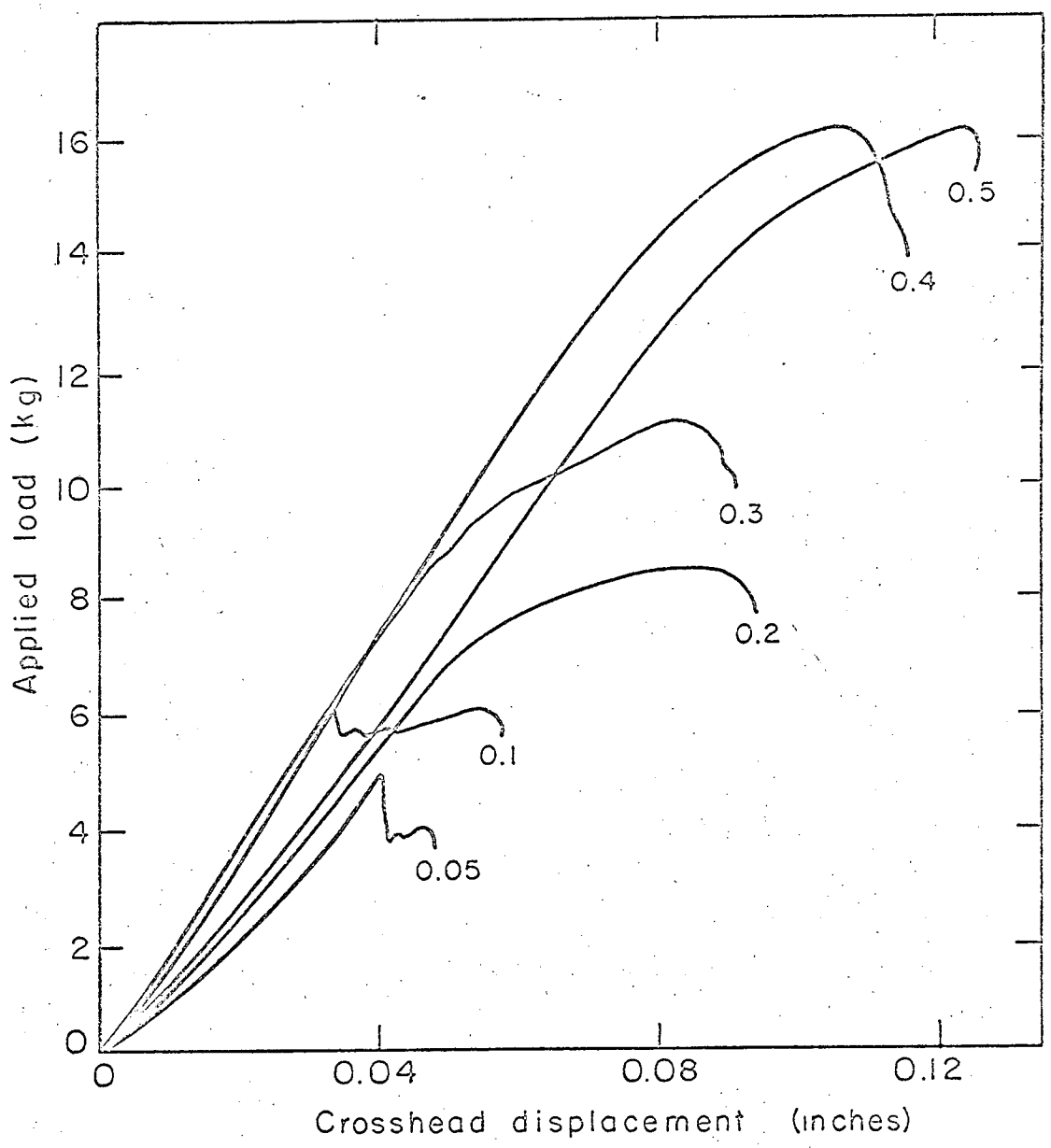


FIGURE 6.

MUB-10459

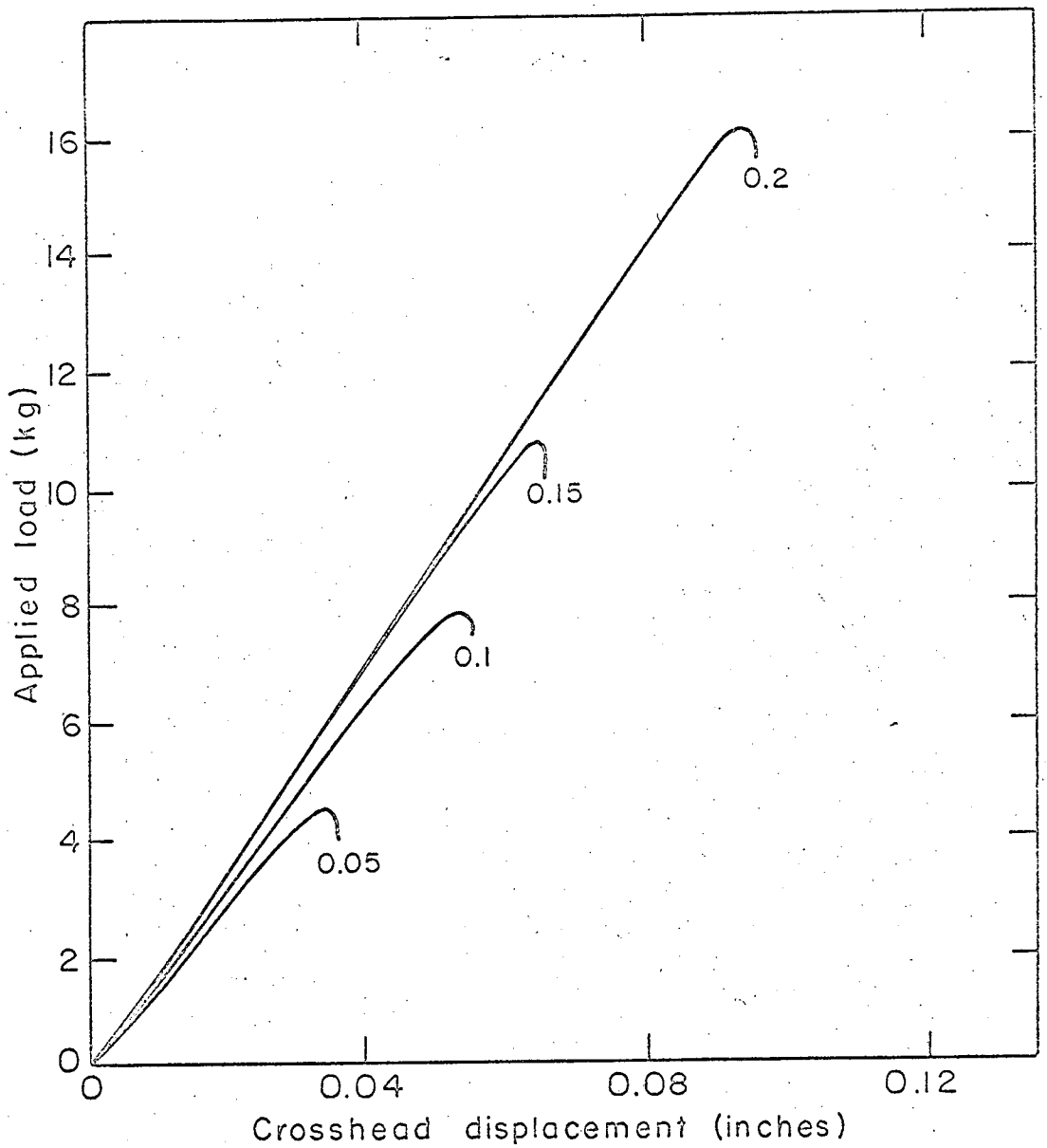


FIGURE 7.

MUB-10460

ZN-5570

Fig. 8

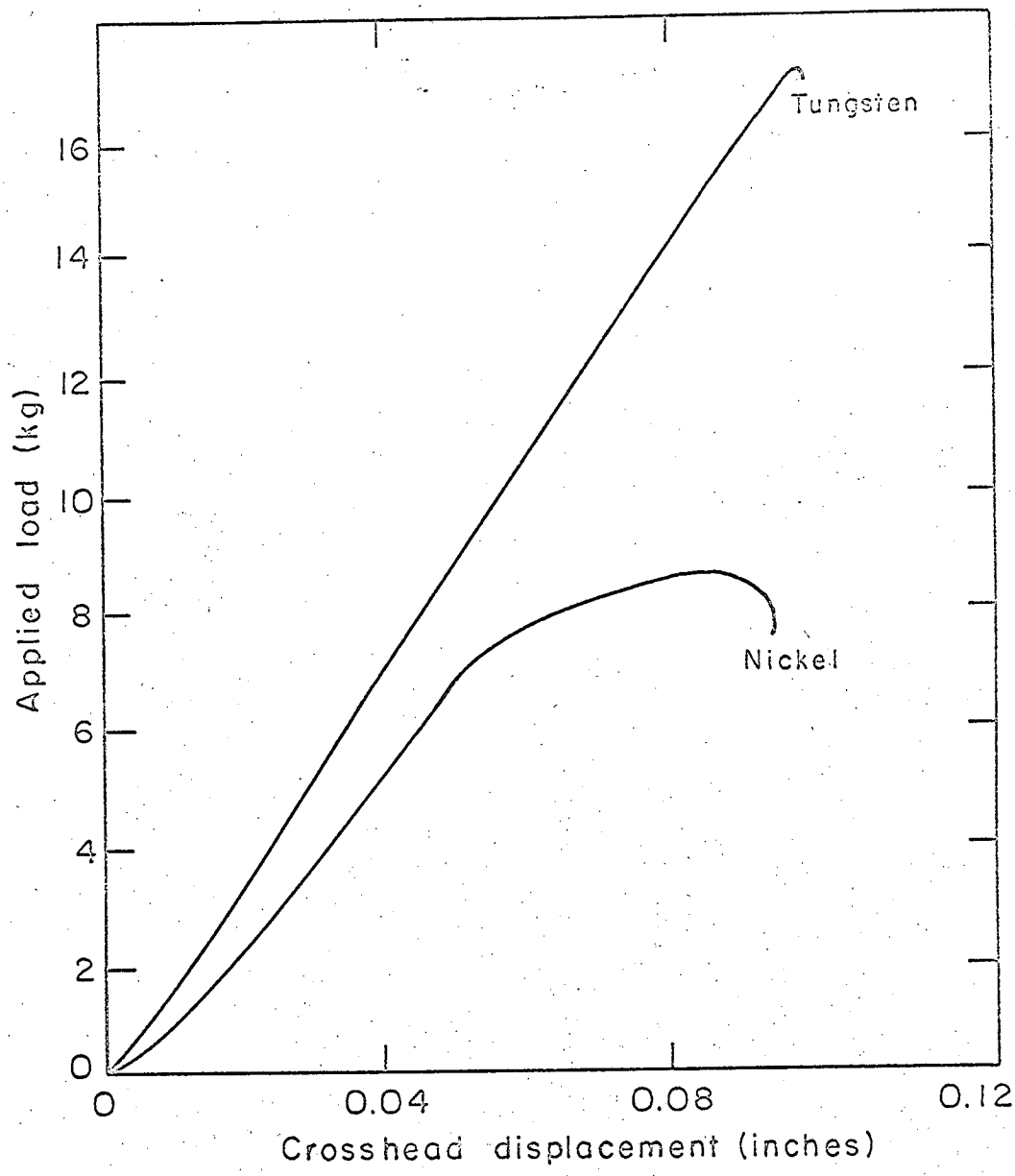


FIGURE 9.

MUB-10461

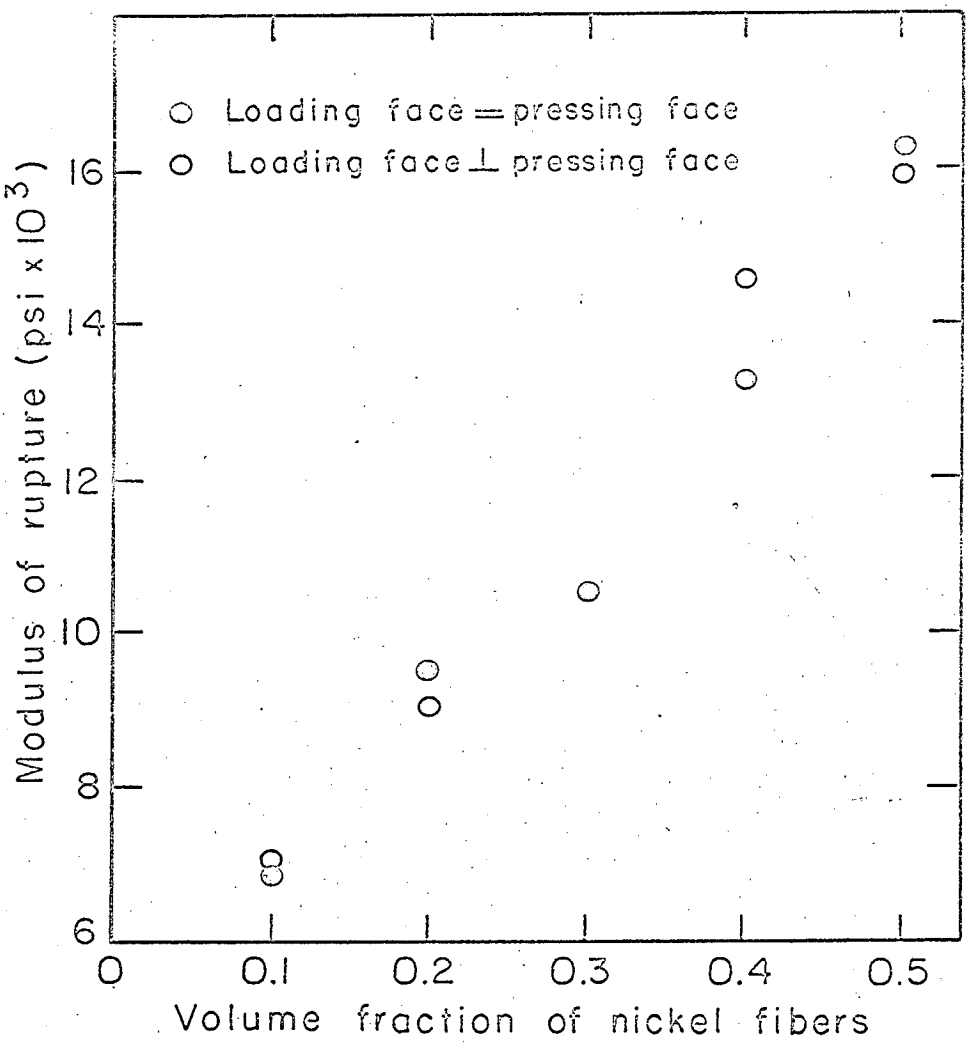


FIGURE 10.

MUB-10435

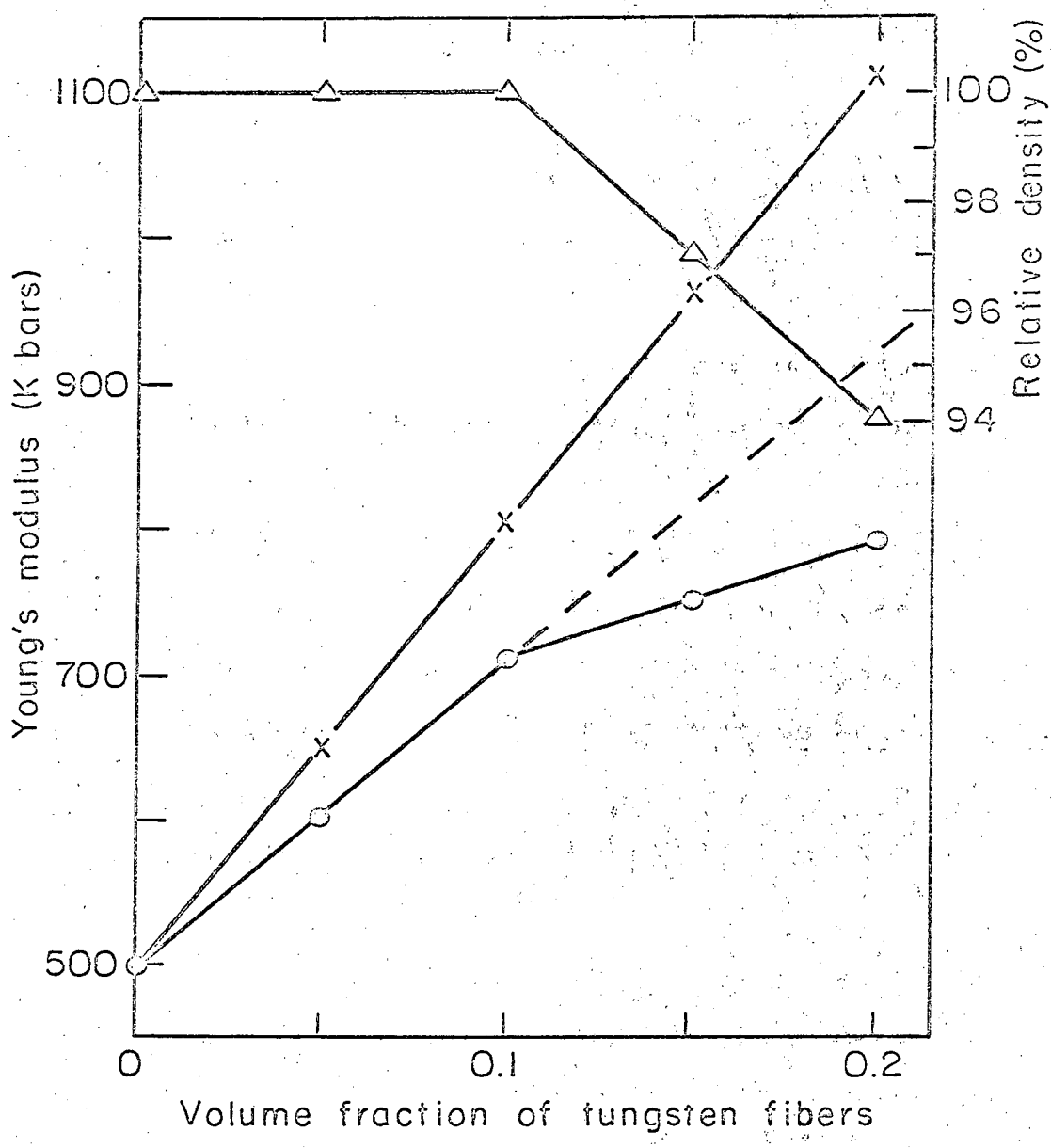


FIGURE II.

MUB-10436

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