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THE DENSIFICATION OF MOLYBDENUM AND MOLYBDENUM ALLOY POWDERS USING HOT ISOSTATIC PRESSING

J. BARRANCO

I. AHMAD

S. ISSEROW

R. WARENCHAK

AUGUST 1985

US ARMY ARMAMENT RESEARCH AND DEVELOPMENT CENTER
LARGE CALIBER WEAPON SYSTEMS LABORATORY
BENÉT WEAPONS LABORATORY
WATERVLIET N.Y. 12189

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This study was conducted to determine a superior erosion resistant gun barrel liner material with improved properties at higher temperatures. Four categories of powders were examined:

1. TZM spherical containing 0.5 titanium, 0.08 zirconium, and 0.02 carbon (wt. % nominally), balance molybdenum (Mo), produced by REP ((Rotating

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7. AUTHORS (CONT'D)

Saul Isserow
Army Materials and Mechanics Research Center
Watertown, MA 02172

20. ABSTRACT (CONT'D)

Electrode Process), PREP (Plasma Rotating Electrode Process), and PMRS
(Plasma Melted and Rapidly Solidified);
2. Mo reduced 2 and 5 μm;
3. Mo-0.1% cobalt, co-reduced;
4. Mo-5 wt. % alumina (Al_2O_3), dispersion strengthened.

Hot Isostatic Pressing (HIP) densification occurred at 15-30 Ksi, 1300-1600°C,
for 1.5 to 3.0 hours. The TZM REP/PREP powders (220/74 μm) were not fully
densified even at 1600°C, 30 Ksi, 3 hours. Point particle contact prevented
complete void elimination. TZM PMRS powder (24.7 μm) achieved 99 percent of
theoretical density while maintaining a small grain size (10.4 ASTM eq.) Bend
deflection and fracture energies were approximately three times those for PREP
powder at a bend rupture strength of about 120 Ksi. Mo reduced and Mo-0.1% Co
powders showed less (or the same) ductility with increasing HIP temperatures.
Fractures were intergranular with decreased bend rupture and compression
strength. The Mo-5Al_2O_3 powder maintained a fine grain size (13 ASTM eq.),
but with fracture energies usually less than 0.6 in.-lbs. Included are
results from bending and compression testing with metallographic and fracture
mode interpretation.

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ACKNOWLEDGEMENTS

INTRODUCTION

The reason for the study was to arrive at a superior erosion resistant gun barrel liner material with improved properties at elevated temperatures. The powder metallurgy approach using molybdenum and molybdenum alloy powders densified by hot isostatic pressing (HIP) is thought to be one way of achieving this goal. Hot working methods will later be pursued to further increase mechanical properties.

The ideal erosion resistant material to be used as a liner for gun barrels for example, depends on the properties of certain special metals or alloys and on the suitability of the firing conditions for which they will be used. The liner must have resistance to thermal and chemical attack caused by the propellant gases and suitable mechanical properties. Early studies by the National Defense Research Committee (NDRC) found only chromium, molybdenum, tungsten, and tantalum showed promise. The early use of molybdenum as a possible liner material was considered by Palmer (ref 1) and others (refs 2,3). Their first attempt to fabricate a molybdenum liner was from tubes bored from swaged rods, but these tubes soon failed after only a few rounds. It was then realized that unless strength and ductility could be improved, the advantages of molybdenum as a liner material would be lost. Researchers found

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[1] F. Palmer, "Experiments on the Melting of Molybdenum," OSRD 6496, Report No. A-425, Climax Molybdenum Co. and Westinghouse Electric and Manufacturing Co. Inc., February 1946.

[2] "Investigation of Gun Erosion at the Geophysical Laboratory," Vol. I, July 1941 to July 1943, OSRD 3448, Report No. A-263, Geophysical Laboratory, CIW, March 1944.

[3] "Investigation of Gun Erosion at the Geophysical Laboratory," Vol. II, July 1943 to December 1943, OSRD 3449, Report No. A-264, Geophysical Laboratory, CIW, April 1944.

the inherent brittleness of the commercially available material was unsuitable for liner fabrication as described, and thus a two-stave helical twisted composite liner was developed (refs 4,5). This allowed better control of properties to be realized during fabrication and improvement of circumferential ductility. Twisting the well-fibered bars of wrought molybdenum resulted in turning the axially elongated grains towards the circumferential direction. This improved the normally weak transverse strength usually associated with uniaxially worked molybdenum bar stock.

The successful deformation of molybdenum was found to depend on a working schedule which first produced a recrystallized grain structure above 1200°C and then converted the rather weak microcyrstalline grains to a structure with larger uniform grains. Further working was accomplished by balancing the amount of reduction (thickness) with descending temperatures to 1000°C as the lower limit. A two-stave twisted 0.50 cal liner of molybdenum-0.1% cobalt alloy with a 0.25-inch wall thickness was produced and withstood over 2000 firings with no evidence of powder gas erosion on the bore surface. However, some unwanted effects were observed after firing. Longitudinal cracking was due to insufficient transverse strength and ductility which then permitted the grains elongated by working to split apart. Spalling occurred along the edges of the seams. Swaging of the lands was due to the lack of hot hardness to withstand engraving stresses, while plastic flow of metal towards the muzzle

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[4]N. H. Smith, "The Behavior of Gun Liners and Coatings Tested Under Conditions of Hypervelocity," OSRD 6475, Report No. A-404, Franklin Institute, October 2, 1945.
[5]P. H. Brace, "Development of Molybdenum for Gun Liners," OSRD 6495, Report No. A-424, Westinghouse Electric and Manufacturing Co. Inc., February 1946.

end of the liner caused bore constrictions sometimes resulting in termination
of the test firing schedule.

Molybdenum can be hardened not only by cold working below the recrystal-
lization temperature, but also by alloying. Cobalt up to two percent,
tungsten from five to twenty percent, and nickel up to seven percent (by
weight) were looked at in earlier studies (ref 6), at which time the alloy
known as TZM (containing titanium and zirconium) was not perfected. The early
attempts to produce billets for making these stave type liners used powder
metallurgy compacts as the only starting material. These powder compacts were
mechanically pressed and sintered, but later hydrostatic pressing was used.
This allowed uniformity of pressing to occur without the formation of
incipient cracks. These cracks frequently opened up rapidly under firing
conditions when mechanical pressing was used in fabricating the starting
billet from which the stave liners were rolled.

The advent of HIP has proved a further improvement in the consolidation
of powder metallurgy compacts. This process was developed at Battelle's
Columbus Laboratory in the mid-1950's (ref 7). The first use of the method
was a diffusion process for cladding nuclear fuel elements. Using HIP for
powder consolidation was a natural outgrowth of the process. The first
application for producing an integral form from powdered metals was for

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[6]J. W. Marden, "Fabrication of Molybdenum as a Gun Liner Material," OSRD 6494,
Final Report No. A-423, Westinghouse Electric and Manufacturing Co. Inc.,
October 31, 1945.
[7]H. A. Saller, S. J. Paprocki, R. W. Dayton, and E. S. Hodge, U.S. Patent No.
687,842 and Canadian Patent No. 680,160, February 18, 1964.

the TZM arc-cast wrought and the pressed and sintered material received as bar stock. A fine grain size was preserved when the PMRS powder was HIP densified in contrast to large grains which were found along with an oriented microstructure for the arc-cast and wrought bar stock. Grain growth occurred for the pressed and sintered bar stock with increasing HIP temperatures. The small grain size retained by the PMRS powder could be important when hot working is anticipated following HIP to further improve the mechanical properties.

The Mo-0.1% Co co-reduced powder HIP densified at successively higher temperatures from 1300° to 1500°C produced excessive grain growth with reduced mechanical properties. An intergranular fracture mode occurred for all the HIP temperatures used. An agglomeration problem was apparent when an HIP container was sectioned following HIP. The agglomeration problem evidently occurred when the cans were filled with powder prior to HIP and later caused cracking because of the differential shrinkage rates occurring within the container during HIP.

The blending of alumina with the hydrogen-reduced molybdenum powder (5 μm average particle size) was highly effective as a grain growth inhibitor with HIP temperatures as high at 1600°C. The powder blend consisting of 5 wt. percent alumina (0.05 μm particle size) after HIP resulted in a brittle material and with low fracture energies. A binder phase of a composition compatible to both the molybdenum and alumina powders could have the effect of modifying the bonding achieved at the particle interfaces with improved strength and ductility. The understanding of the bonding and wetting of ceramics requires a more detailed study than was attempted here for this

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A twin to the spherical TZM powder described above was also examined and designated SX-208. The composition was molybdenum with 3.5 wt. percent cobalt. Unfortunately, the powder quality was poor and contained internal porosity on a gross scale. Limited funding prevented pursuing refinement of the plasma parameters to eliminate these shortcomings.

Since the TZM spherical powder has been proven as a viable candidate material for further working by extrusion, it was decided to improve its overall quality. This will require improvement in chemical homogeneity, elimination of satellite formation sometimes seen, and improved cleanliness with removal of foreign contamination.

SUMMARY

The TZM spherical powders produced by the rotating electrode process did not achieve acceptable consolidation using the HIP process. This was true for both REP and PREP powders even when the PREP powder was preferentially screened below 200 mesh (74 µm average particle diameter) and only using that portion for HIP. The mean particle diameter resulting from the rotating electrode process in making the TZM powder was large (220 µm), and this method inherently does not allow wide variation of process variables to arrive at finer particle sizes in the amounts desired.

The TZM powder produced by the Plasma Melting and Rapid Solidification Method (PMRS) was spherical with an average particle size of 24.7 µm. This powder HIP densified well and properties in both bending and compression were better than for the other alloy powders tested. Values were lower compared to

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FUTURE ACTIVITY

The investigation of microstructures and properties in bending and compression for materials densified by the HIP method as shown in Table I, was completed in order to choose candidate materials whose properties could be substantially improved by working the densified compacts following HIP. The TZM (GTE) spherical powder designated SX-207 was chosen as one of the candidate materials. Comparison of this densified powder to the arc-cast TZM (P) material in bending is seen in Figure 29 for both notched and unnotched bars tested. Figure 30 shows compression tests for both materials. The TZM (P) material reached higher mechanical properties in both bending and compression, but other factors should be taken into account when a cylindrical hollow shaped part is contemplated as the final configuration. This would require that transverse ductility be considered, whereas alloys produced by arc-melting and later worked, often show a high degree of anisotropy. This difficulty will be overcome hopefully using the powder metallurgy approach. It is interesting to note that after exposure to HIP temperatures of 1500° and 1600°C, the hardness of SX-207 remained almost constant and about the same as the arc-cast TZM hardness of 20.0 Rc. The high apparent density of this powder (5.32 g/cc) makes it desirable along with its spherical particle shape which insures free flowing characteristics and is less likely to cause agglomeration or result in bridge formation even at an average particle size of only 24.7 µm. The resulting hardness appears adequate to prevent swaging of the rifling at the bore surface which must be maintained to insure a gas seal and to maintain accuracy during service.

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The properties of two commercially prepared solid bars of TZM were designated TZM (P) and TZM (GTE) as shown in Table II. The TZM (P) material was arc-cast and wrought to form a four-inch diameter billet. The TZM (GTE) material was in the form of 1-1/4-inch diameter bars from pressed and sintered powder, but prior to working as would normally be done consistent with further commercial practice. Figures 25 and 26 show comparison of properties in bending and compression, respectively. The notched properties in bending and compression testing can be used for exact comparison, but not the unnotched properties in as-received condition. A 0.357-inch diameter smooth round bar was used in bend testing for the TZM (GTE) as-received material as a last minute substitute for a 0.394-inch square bar due to scarcity of material. This inconsistency should be noted. Compression testing in as-received condition resulted in similar behavior between arc-cast TZM (P) material and TZM (GTE). TZM (GTE) notched bars showed improvement in ductility with increasing HIP temperatures as shown in Figure 27.

The use of alumina at 5 wt. percent to control grain growth is demonstrated in Figure 16, where a small grain size persisted even at a HIP temperature of 1600°C. The 5 μm reduced molybdenum powder was used as the matrix phase. Reductions in bend rupture strength, bend yield strength, and fracture energy were observed with increasing HIP temperatures to 1600°C as shown in Figure 28 for both smooth and notched conditions. The compacts were extremely brittle, and because of this, material for making compression specimens was not available after numerous specimens fractured during machining. Increasing the alumina phase, Figure 18, brought about agglomeration and a large amount of void formation and was not further purused.

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The Mo-0.1% Co co-reduced powder, when HIP densified at successively higher temperatures of from 1300° to 1500°C, actually resulted in lower mechanical properties being obtained. This was evident for tests conducted in bending and compression; see Figures 21 and 22. A decrease in hardness is usually observed with increased HIP temperature, but with improved ductility and increased fracture energy. This was not fully the case in bend tests performed and shown in Figure 21. Fracture energies for unnotched bars tested varied little. Intergranular fracture mode occurred with all HIP temperatures used for densification with rapid grain growth observed at 1500° and 1600°C, Figures 7 and 8, and likely was detrimental to final properties obtained. This powder showed evidence of agglomerating when canned prior to HIP densification. Packing did not appear uniform and this resulted in different amounts of shrinkage in certain regions leading to weakness and cracking as shown in Figure 6. The packing also contributed to poorer mechanical properties than desired.

The two molybdenum reduced powders of 2 μm and 5 μm indicate the smaller particle size powder resulted in the higher bend yield, rupture strength, and fracture strength. This is illustrated in Figures 23 and 24 for HIP conducted at 1400° and 1500°C. The oxygen content of the 2 μm powder was less than 2900 PPM while the oxide content of the 5 μm powder was less than 700 PPM. It is possible that the higher oxide content of the 2 μm powder acted to prevent rapid grain growth from occurring when compared to the 5 μm powder seen in Figure 10. This would allow a higher hardness to be maintained as indicated in Table II along with higher strength values observed.

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three hours did not produce the result desired. See Figure 18. In Figure 18a
the powder mixture before consolidation was visably agglomerated, while in
Figure 18b the polished surface of a densified specimen indicates a non-
uniform dispersion of both powders with a large amount of porosity present.
The fracture surfaces shown in Figures 18c and 18d were obtained from bars
which fractured while being machined into bend bar specimens. These SEM
photographs reveal poor particle bonding present, resulting in low fracture
energy values.

DISCUSSION

The TZM spherical powder produced by the REP and PREP process, even when
preferentially screened to an average particle size of 74 μm, did not densify
as shown in Figures 2 and 3. The TZM (GTE) spherical powder designated as
SX-207 with an average particle size of 24.7 μm reached theoretical density
and achieved better mechanical properties. The results of bend tests show
this in Figure 19 for both powders. Compression tests run on both HIP
densified powders are shown in Figure 20, however, the superiority of the
SX-207 powder is not readily apparent. The difficulties experienced with the
TZM REP and PREP powders in attaining full density are likely due to the large
numbers of voids observed even after HIP at 1600°C, 30 Ksi, for four hours.
The possibility of surface particle contamination upon processing is possible
as a hindrance to densification, but the gross average particle size of the
one compared to the finer particle size of the other (e.g., 220 vs. 24.7 μm)
was most likely the deciding factor in determining the difference in
densification obtained.

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as a result of bend tests performed after HIP are shown in Figure 14. They
indicate cleavage as the fracture mode. The TZM (P) material was received in
the form of bar stock from commercially arc-cast and wrought TZM with a
diameter of 4.0 inches after working. Bend specimens were taken from the
transverse direction. The microstructure of this material, Figure 15,
indicates a worked structure with cleavage as the fracture mode not unlike
that seen previously.

Molybdenum-Oxide Powder Mixtures

The use of a dispersoid as a means to control grain growth led to the
mixing of 5 wt. percent alumina (Al_2O_3) with molybdenum powder. The size of
the alumina powder was 0.05 μm (Baikalox) and the molybdenum reduced powder
(G.E.) was 5 μm. The alumina behaved as a grain growth inhibitor as seen in
Figure 16. While the HIP temperatures were increased from 1400 to 1600°C at
15 to 30 Ksi up to three hours, grain size remained almost constant. The
density increased from 9.33 to 9.50 g/cc while hardness decreased from 34.8 to
33.2 Rc. Tensile bars tested indicated an ultimate strength of 35 to 36 Ksi
with little ductility observed. Bend tests indicate that yield and rupture
strengths are reduced in general with increasing HIP temperatures.
Nonuniformity of the alumina dispersion is evident in Figure 17b. This
illustrates the problem more clearly using SEM compared to the etched and
polished microstructures in Figure 16. Figure 17d shows a type of inter-
granular fracture developed when HIP temperatures were raised to 1600°C.
Success with alumina in maintaining a small grain size using 5 wt. percent was
extended to 60 wt. percent. A specimen HIP densified at 1600°C and 30 Ksi for

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being pursued in P/M research to control grain size. Properties obtained for the 2 μm powder densified at 1500°C are clearly superior to those of the 5 μm powder as listed in Table II. Examination of fracture surfaces from bars HIP densified at 1500°C indicates intergranular fractures occurred in both, but the grain size difference is clearly evident per Figure 11.

Solid Bars - TZM

The materials investigated in Table I were mostly in powder form. There are two exceptions. One is the TZM (GTE) pressed and sintered bars 1-1/4 inches in diameter which were later HIP densified, and the other is the TZM (P) 4-inch diameter bars used for testing without any HIP treatments. These two materials were selected as a guide to compare the effect of HIP densification using canned powders and to compare properties attained from both. The microstructure for the TZM (GTE) bars as-received, shown in Figure 12, appears much different after exposure to HIP temperatures, shown in Figure 13, where exposure to successively higher temperatures and times is evident. This exposure has annealed the original structure causing an increase in grain growth. The as-received bar of TZM (GTE) designated as A(SR) was machined as a smooth round bar. When tested in bending it had an indentation on the circumference caused by the small diameter dowel pin notching the specimen under load at mid-span. This pin, in effect, notched the smooth round bar preventing an exact comparison with the smooth square bend bars tested. The smooth round bar has a higher yield and rupture strength compared to TZM (P), but has lower fracture energy influenced partly by higher hardness and the notching effect mentioned along with unequal cross-sectional area compared to the square bars normally used. Fracture surfaces obtained from notched bars

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variation resulted in a grain growth phenomenon in a specific area of large grains surrounded by much smaller grains. The result was a crack running through this area to the I.D. of the can. The results of increasing HIP temperatures on densifying the powder are shown in Figure 7. Here we see a dramatic increase in average grain size from 1300° to 1500°C. Figure 7 also shows polished and etched microstructures. Fractographs of the broken bend bars in Figure 8 indicate intergranular failure occurred consistently. However, a rounding of the fracture surfaces at 1500°C indicates diffusion was likely enhanced. The rupture strength declined uniformly with increasing HIP temperatures using smooth bars with notched values at 28 and 17 percent of the smooth bar values at 1400° and 1500°C. Fracture energies fell to extremely low values, e.g., 0.1 in.-lb for notched bars compared to 4 or 5 in.-lbs for unnotched bars.

Reduced Powders

Fine homogeneous powders of TZM were not readily available except for the SX-207 spherical powder acquired later in this study. Since Mo reduced powder in the 2-5 µm range could be easily acquired, it was decided to compare pure Mo powder HIP densified using similar parameters. The reduced molybdenum powder was acquired from G.E., Cleveland, OH in the 2 and 5 µm particle size shown in Figure 9. Figure 10 reveals the effect of HIP parameters at 1400 and 1500°C for both powders. A much larger grain size due to grain growth is apparent for the 5 µm powder but may not be wholly dependent on starting particle size. The oxygen content of the 2 µm powder was greater than for the 5 µm powder (2900 vs. 700 PPM). This could have inhibited grain growth by pinning the prior particle boundaries by the oxide. This type of approach is

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better, but porosity was still evident as shown in Figure 3. This spherical powder did not lend itself to HIP densification even at 1600°C and 30 Ksi for three hours duration. Void formation and prior particle boundaries were evident. Bend deflection and rupture strength improved using the higher HIP temperatures, but values were far less compared with the properties attained using the (GTE) spherical powder.

The spherical TZM powder prepared by Sylvania (GTE) and designated as SX-207 had a mean particle diameter of 24.7 µm. The powder surfaces and microstructure in Figure 4 indicate small grains present within each powder particle. Figure 5 illustrates the fracture surface and microstructure of this consolidated powder. The TZM (PREP) samples consisted of large particles mostly semi-bonded shown in Figure 3a, while the SX-207 powder indicates only remnants of prior particle boundaries present in Figure 5a. Figure 5b shows a few isolated voids existing in the polished and etched surface. The fracture energy is higher compared to the TZM (PREP) powder with HIP at 1600°C outlined in Table II.

Co-Reduced Powder

An interesting powder was supplied by AMAX Corp., Greenwich, CT. This was the co-reduced Mo-0.1% Co powder produced by chemical reactions with the intent of insuring a homogeneous powder as the final product. Elemental mixing of Mo and Co powders examined prior to this study proved unsatisfactory and showed segregation upon sintering. The powder supplied had a mean particle size of less than 1.7 µm. This powder with a low apparent density of 2.36 g/cc sometimes segregated during the canning process. This was evident after HIP as seen in the microstructures in Figure 6. Packing density

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(LVDT) gage to measure bend deflection. Tests conducted on these bend bars resulted in values for bend yield and bend rupture strength as well as flexural modulus (not corrected for machine compliance), maximum deflection, and energy to fracture data.

The compression specimens measured 3/8-inch in diameter and one inch in length. These specimens allowed the yield and modulus (not corrected for machine compliance) to be measured in compression. They also provided a density measurement to be made based on weight per unit volume, disregarding internal porosity. Finally, hardness tests were made on all specimens using the Rockwell C scale Brale diamond indenter.

Metallographic evaluation was done using the scanning electron microscope (SEM) and using standard polishing and etching procedures.

RESULTS

The categories of materials investigated in this study are shown in Table I. The experimental results of testing performed on these HIP densified samples are shown in Table II.

Spherical Powders

The spherical powder received from Nuclear Metals Inc. of Concord, MA initially had a mean particle diameter of 220 μm but was further screened to yield 200 mesh powder (74 μm) that was used for final HIP densification shown in Figure 1. The first attempt to densify the rotating electrode type powder (REP) without screening was met with little success as shown in Figure 2. The second batch of powder received produced by a similar process using the plasma electrode rotating process (PREP) and preferentially screened densified

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which was smaller (e.g., 24.7 μm) compared to the 220 μm mean (average) particle size diameter produced by the REP or PREP process.

The remaining powders examined were either the hydrogen-reduced type supplied by G.E., Cleveland, OH, or the chemically co-reduced type in the case of molybdenum with 0.1 percent cobalt (Mo-0.1% Co) supplied by AMAX Corp., Greenwich, CT.

The characteristic properties of these powders obtained using the HIP method of densification were used to select candidate materials that could be further improved by deformation following HIP. Hot extrusion was chosen as the best method to form a cylindrical shape that could be made into a liner for gun barrel application.

EXPERIMENTAL PROCEDURE

Fabrication of all HIP specimens was done by I.M.T. of Andover, MA using an inert gas (argon) to accomplish densification of the canned powder specimens. Pressures ranged between 15 and 30 Ksi from one and one-half to four hours at temperatures between 1300 and 1600°C. The cans made to contain the powder were typically titanium and in one instance, stainless steel evacuated and sealed prior to HIP.

The HIP specimens normally measured 3/4-inch in diameter and up to 8 inches long. The outer cans were machined off after pressing and the compacted specimens were sectioned and cut up for testing purposes.

The bend bars (ASTM E-23) were machined with essentially the same dimensions as a Charpy bar with and without a notch. The slow bend tests performed on these bars were done on an Instron universal testing machine at a crosshead speed of 0.02 in./min with a low voltage differential transformer

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degree of anisotropy, whereby the transverse ductility is generally very poor.

Recent developments of the rapid solidification technology (RST) are of unique significance because this method can result in powders which are small, spherical, and homogeneous in composition. When these powders are densified and worked, they often produce properties which are far superior to cast or wrought alloys. This has been observed for aluminum and titanium alloys and for some steels and superalloys. Our study examines spherical powders of TZM produced by two methods. The larger spherical powders were produced by either the rotating electrode process (REP) or the plasma rotating electrode process (PREP). The REP method (ref 11) uses a water cooled tungsten-tipped cathode to strike an electric arc to the rotating molybdenum anode from which molten droplets are centrifugally flung into a vacuum or inert gas collector. The PREP method is similar, but now a helium plasma arc gun replaces the tungsten-tipped cathode to prevent tungsten pick-up which was later found to be a powder contaminant and to insure more uniform melting. Molybdenum powders made by both methods were used and supplied by Nuclear Metals, Concord, MA. The second type of spherical powder examined was produced at GTE Products Corp., Towanda, PA (ref 12). Here a plasma gun was used to remelt the powder by feeding it through a plasma arc and cooling rapidly in an inert gas. This method, Plasma Melting and Rapid Solidification (PMRS), produced TZM powder

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[11] P. R. Roberts, "Commercial Atomization by the Rotating Electrode Process," Atomization Processes: Current and Future, Int. P/M Conference, Toronto, Canada, June 17, 1984, pp. 51-78.

[12] R. F. Cheney, "Plasma Melted and Rapidly Solidified Powders," GTE Products Corp., presented at the Annual Meeting of the Materials Research Society, Boston, MA, 14-17 November 1983.

beryllium (refs 8,9), a difficult to fabricate material. Later the Air Force
and Battelle Columbus (ref 10) used the economic advantage of HIP in
consolidating titanium alloy powders into near net shapes. This resulted in
properties essentially equivalent to the forged material.

The past twenty years has seen considerable progress overall in both
powder metallurgy and metal working technology. We have seen better powder
working and characterization techniques and better densification processes
such as liquid phase and activated sintering, along with hot isostatic
pressing and hot hydrostatic extrusion of powder metallurgy (P/M) parts. TZM
is about the only major alloy of molybdenum which is commercially available in
a variety of geometric forms. It is produced by both vacuum arc-melting and
by powder consolidation technique as a final manufacturing method. TZM
nominally consists of 0.5 percent titanium, 0.1 percent zirconium, 0.02
percent carbon, and the balance molybdenum. The P/M method consists of mixing
the chemically reduced powders of the alloying elements needed, then
compacting them, followed by sintering and working at elevated temperatures.
Cold working is needed in both methods of production to produce strength and
ductility in an alloy that is normally brittle with poor strength values.
Alloys produced by arc-melting and later worked or swaged often show a high

[8]E. S. Hodge, P. J. Gripshover, and H. D. Hanes, "Properties of Gas Pressure
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[10]"Hot Isostatic Pressing of Titanium 6Al-4V," Titanium Science and
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preliminary work.

The characteristics of these powders and the properties obtained using the HIP method of densification will be used as a guide to select candidate materials that can be further improved by hot working.

CONCLUSIONS

1. The TZM powders produced by the rotating electrode process were inherently coarse (220 μm) and did not achieve acceptable consolidation using HIP.

2. The TZM powder produced by the plasma melting or PMRS method was finer (24.7 μm) and reached theoretical density with reduced grain growth and better mechanical properties in bend tests. Improvement in chemical homogeneity should result in higher mechanical properties.

3. Reduced molybdenum powders of 2 and 5 μm subject to HIP indicated intergranular type fractures resulting in low fracture energy values.

4. Mo-0.1% Co (by wt.) powder prepared by co-reduction was fine (1.7 μm) and tended to agglomerate. Grain growth was apparent and intergranular fractures with extremely low fracture energies were recorded.

5. Blending of alumina (5% by wt.) with hydrogen-reduced molybdenum powder was highly effective as a grain growth inhibitor. However, it resulted in a brittle material with low fracture energies.

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    Pressure Consolidated Beryllium Powder," Beryllium Technology 2, paper
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9. H. D. Dumas, P. J. Gripshover, and E. S. Hodge, "Fabrication of Complex
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10. "Hot Isostatic Pressing of Titanium 6A1-4V," Titanium Science and
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11. P. R. Roberts, "Commercial Atomization by the Rotating Electrode
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    Conference, Toronto, Canada, June 17, 1984. pp. 51-78.

12. R. F. Cheney, "Plasma Melted and Rapidly Solidified Powders," GTE
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TABLE I. POWDERS USED FOR HIP EVALUATION

Material	Manufacturers	Type Powder	Particle Size Average (μm)	Apparent Density (g/cc)
Series I Mo(95)Al$_2$O$_3$(5)	GE/Baikalox	reduced-irregular	5.0/0.05	2.46
TZM (REP)	Nuclear Metals	spherical	220	6.22
TZM (GTE)	Sylvania	pressed and sintered bar	-	10.0
Al$_2$O$_3$	Baikalox	irregular	0.05	0.18
Series II Mo-0.1% Co	AMAX	co-reduced/irreg.	1.7	2.36
Mo (2 μm)	GE	reduced-irregular	2.0	1.53
Mo (5 μm)	GE	reduced-irregular	5.0	2.58
TZM (PREP)	Nuclear Metals	spherical	74.0	6.33
Mo(60)Al$_2$O$_3$(40)	GE/Baikalox	reduced-irregular	5.0/0.05	1.62
TZM (SX-207)	Sylvania	spherical	24.7	5.32
TZM (P)	AMAX	arc-cast/wrought bar	-	10.10

TABLE II. R.T. PROPERTIES OF HIP SPECIMENS*

Material	Code	HIP Temp °C	Hardness Rockwell Rc	Density g/cc	Three-Point Slow Bend Test					Compression Test	
					Yield Ksi	Rupture Str Ksi	Flex Mod mKsi	Max Defl mils	Fracture Energy in.-lb	YS 0.2% Ksi	Modulus mKsi
TZM (PREP)	11(N)	1500	8.6	9.6	73.6	73.6	22.9	4.3	2.6	61.5	10.5
	12(N)	1600	15.3	10.0	73.1	73.1	29.8	3.2	1.9	65.0	17.0
	8(S)	1600	15.8	9.9	.53.6	111.5	13.2	22.1	47.7	65.0	15.3
TZM (SX-207)	15(N)	1500	20.0	10.1	85.8	85.8	21.8	5.2	3.6	59.9	9.7
	16(S)	1500	20.0	10.1	69.3	127.0	9.8	35.2	87.4	59.9	9.7
	17(N)	1600	18.0	10.1	79.7	79.7	21.7	4.9	3.2	53.4	13.0
	18(S)	1600	18.2	10.1	61.3	126.0	9.7	57.1	145.3	53.4	13.0
Mo (2 μm)	A	1400	20.7	10.1	-	-	-	-	-	98.5	34.0
Mo (5 μm)	B(S)	1500	16.9	10.1	100.0	100.0	17.0	6.2	8.1	85.0	16.7
	C(S)	1400	15.2	10.1	61.6	61.6	12.4	5.2	4.2	89.7	20.0
	D(S)	1500	14.8	10.1	42.4	42.4	14.1	3.2	1.7	67.0	14.0
Mo-0.1% Co	16(S)	1300	15.7	10.1	73.2	73.2	14.9	5.2	4.9	88.4	25.2
	21(S)	1400	13.6	10.1	65.3	65.3	14.1	4.9	4.1	94.0	40.5
	32(N)	1400	14.0	10.1	18.3	18.3	19.3	1.3	0.2	73.0	20.3

TABLE II. R.T. PROPERTIES OF HIP SPECIMENS* (CONT'D)

Material	Code	HIP Temp °C	Hardness Rockwell Rc	Density g/cc	Three-Point Slow Bend Test					Compression Test	
					Yield Ksi	Rupture Str Ksi	Flex Mod mKsi	Max Defl mils	Fracture Energy in.-lb	YS 0.2% Ksi	Modulus mKsi
Mo-0.1% Co	43(N)	1500	13.6	10.1	10.0	10.0	17.5	0.8	0.1	58.7	11.6
	54(S)	1500	13.6	10.1	42.3	57.7	12.7	5.5	4.6	57.0	10.4
Mo-5Al$_2$O$_3$	1(SR)	1400^b	38.4	9.3	113.0	113.0	15.0	9.0	5.8	-	-
	2(SR)	1400	38.5	9.3	115.0	115.0	14.0	9.4	6.1	-	-
	3(SR)	1500	34.8	9.3	92.0	92.0	14.0	7.6	4.0	-	-
	4A(N)	1500^a	36.5	8.2	40.0	40.0	17.0	3.0	1.0	-	-
	4B(N)	1500^a	36.5	8.2	47.0	47.0	19.0	3.2	1.3	-	-
	5(N)	1600^c	34.0	8.2	18.0	18.0	19.0	1.3	0.2	-	-
	6(N)	1600	33.2	9.5	23.0	23.0	19.0	1.7	0.3	-	-
TMZ-P (Transverse)	21(N)	-	22.1	10.1	97.9	107.4	25.7	6.0	5.7	90.0	15.5
	22/23(S)	-	22.1	10.1	145.0	194.0	12.9	64.4	276.4	90.0	15.5
TMZ (GTE)	4(N)	-	25.0	10.0	103.5	103.5	20.0	7.0	5.9	86.0	9.4
	A(SR)	-	25.0	10.0	168.0	265.0	9.0	114.3	266.0	86.0	9.4
	B(1)(N)	1400^b	13.8	10.0	92.0	116.0	23.0	8.3	9.2	56.0	12.3

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TABLE II. R.T. PROPERTIES OF HIP SPECIMENS* (CONT'D)

Material	Code	HIP Temp °C	Hardness Rockwell Rc	Density g/cc	Three-Point Slow Bend Test					Compression Test	
					Yield Ksi	Rupture Str Ksi	Flex Mod mKsi	Max Defl in.	Fracture Energy in.-lb	YS 0.2% Ksi	Modulus mKsi
TZM (GTE)	B(2)(N)	1400b	13.8	10.1	91.3	191.0	8.0	375.0	1553.0	56.0	12.3
	2(N)	1500	12.7	10.0	69.0	102.5	14.5	16.2	18.4	-	-
	3(N)	1600a	13.8	10.0	71.0	101.0	12.0	16.1	16.5	51.0	7.6
	4(N)	1600c	10.1	10.0	45.5	96.0	17.5	12.6	12.8	51.0	7.6

*30 Ksi - Three hours 0.394 in Charpy bar (N) notched (S) smooth
0.357 dia smooth round bar (SR)
0.357 dia compression specimen x one inch long

a15 Ksi - 1.5 hours
b30 Ksi - 1.5 hours
c15 Ksi - 3.0 hours

24

(a)
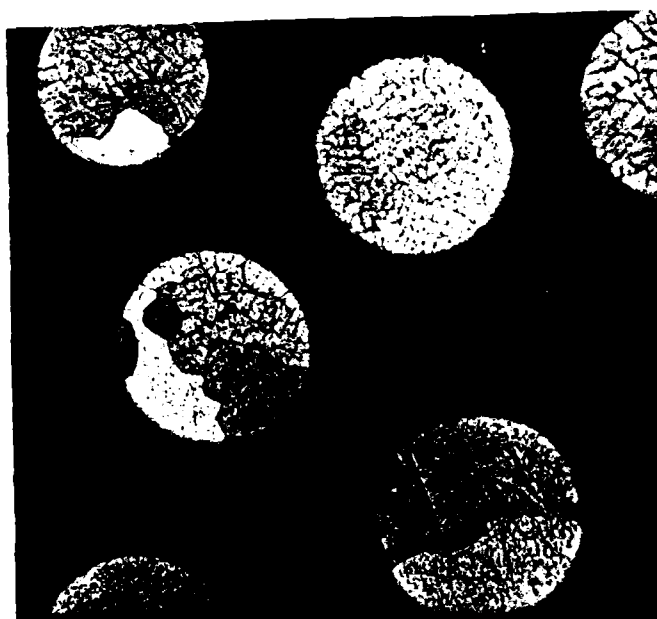
(b)

Figure 1. TZM spherical powder produced by the rotating electrode process (REP) - (a) powder, SEM (300X) and (b) polished and etched microstructure (200X).
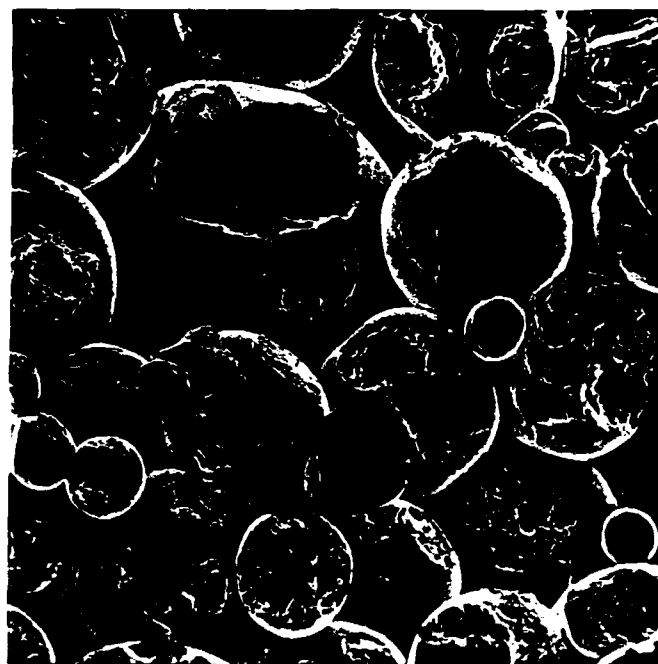
(a)

(b)

Figure 2. TZM spherical unsieved powder (mean particle diameter 220 μm) produced by the rotating electrode process (REP) and HIP densified at 1500°C at 30 Ksi for three hours indicating point contact sintering – (a) tensile bar fracture surface SEM (125X) and (b) polished and etched microstructure (100X).

(a)
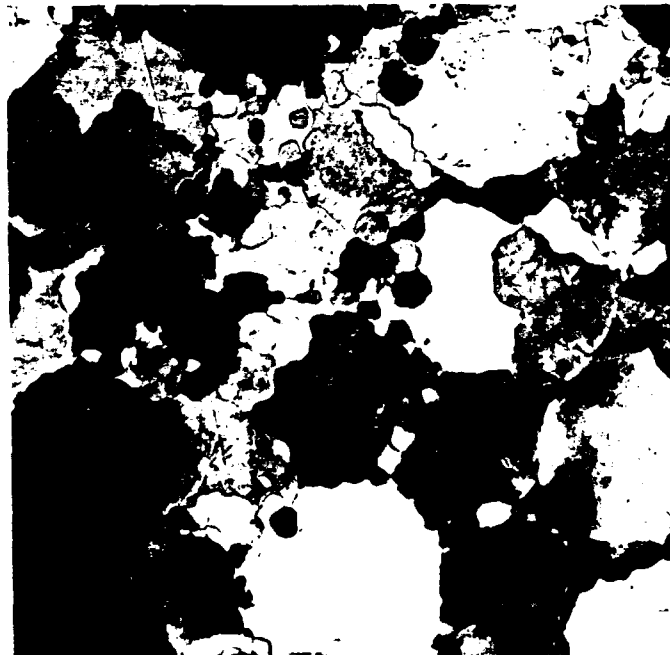
(b)

Figure 3. TZM spherical powder produced by the plasma electrode rotating
process (PREP) and HIP densified at 1600°C at 30 Ksi for four
hours — (a) three-point bend fractograph SEM (300X) and (b)
polished and etched microstructure (200X).
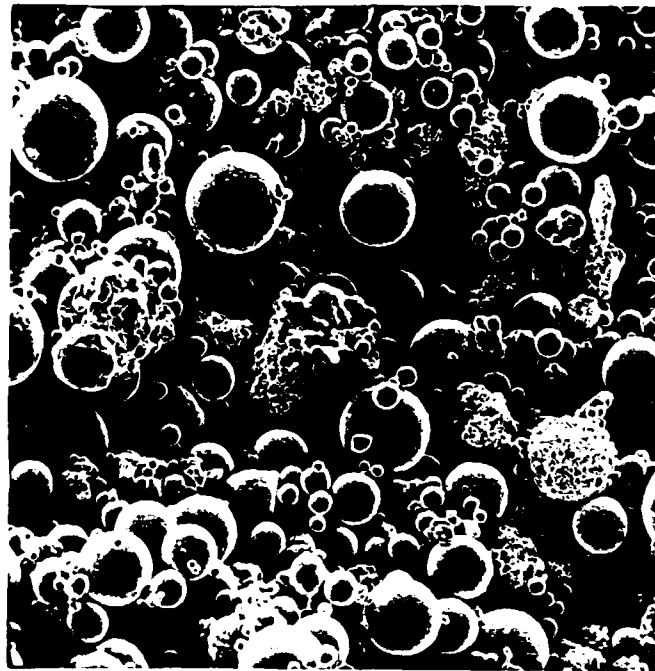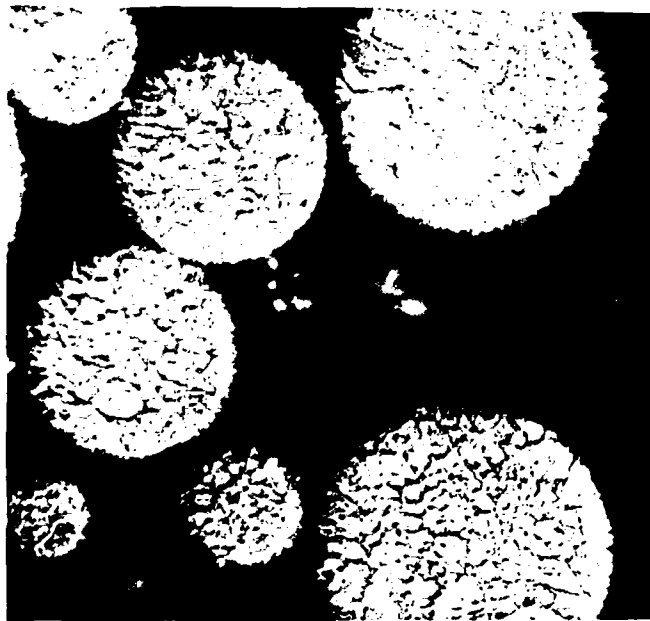
27

(a)

(b)

Figure 4. TZM spherical powder, SX207 (average particle diameter 24.7 μm) produced by the plasma melting and rapid solidification process (PMRS) - (a) powder, SEM (300X) and (b) polished and etched microstructure (1000X).

28
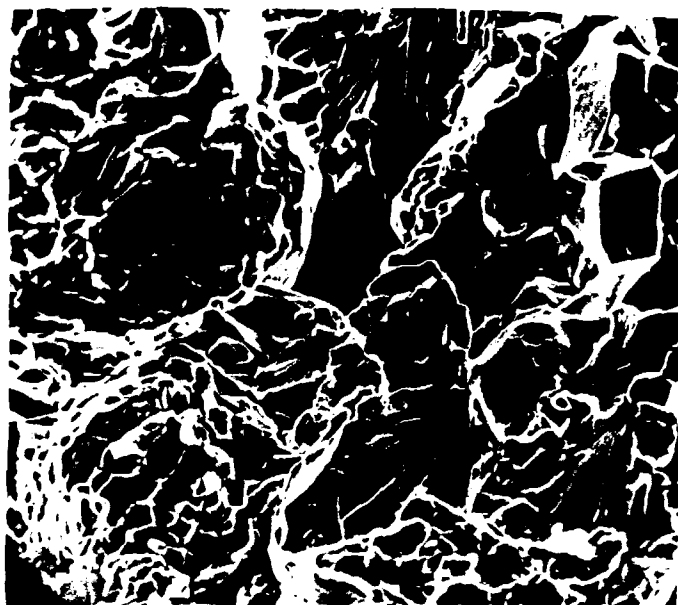
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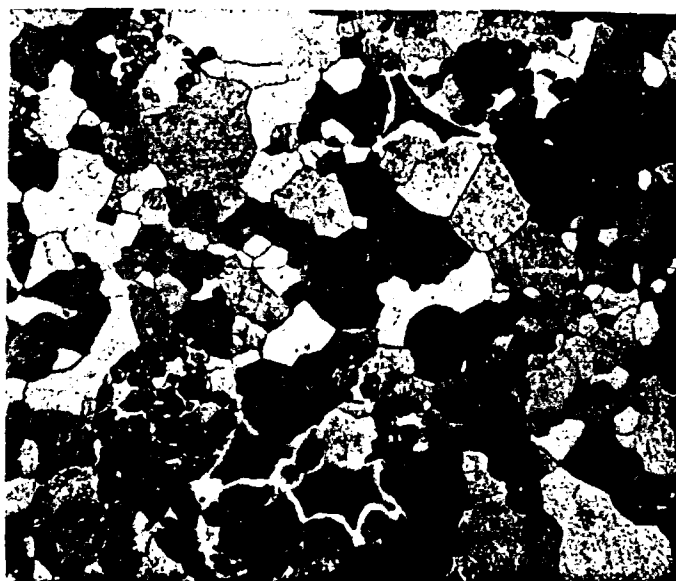
(b)

Figure 5. TZM spherical powder, SX207 (average particle diameter 24.7 μm)
           produced by the plasma melting and rapid solidification process
           (PMRS) and HIP densified at 1600°C at 30 Ksi for three hours –
           (a) three-point bend fractograph, SEM (1500X) and (b) polished
           and etched microstructure (500X).

29

(a)

(b)

(c)
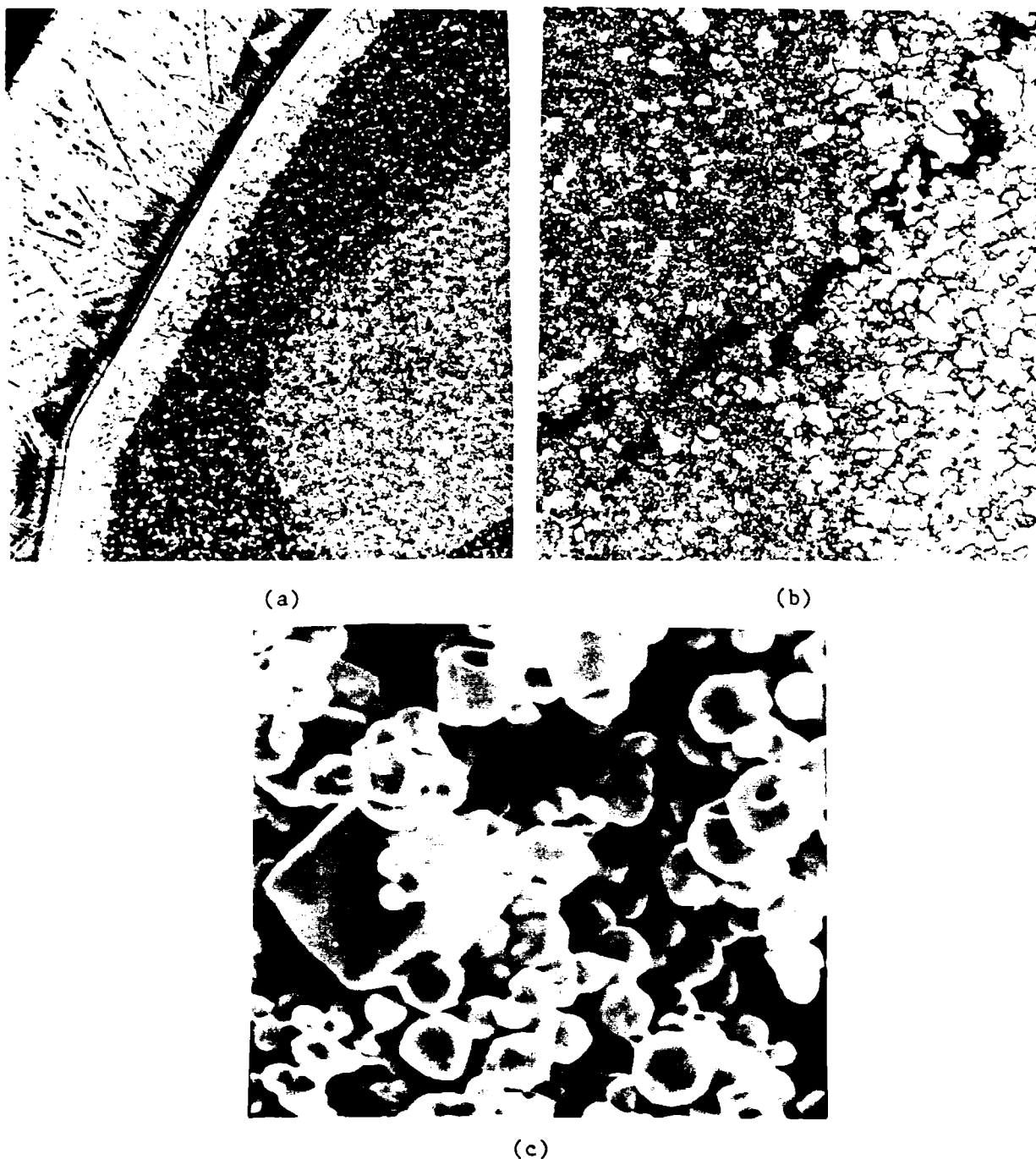
Figure 6. Mo-0.1% Co co-reduced powder of small particle size (1.7 μm) and
low apparent density (2.16 g/cc) showing powder packing
segregation with cracking occurring during the HIP densification
cycle at 1400°C - (a) transverse view across the can diameter
showing macro segregation (20X), (b) variation in grain size
across interface with crack (100X), and (c) powder surfaces, SEM
(4000X).

30

(a)

(b)

(c)
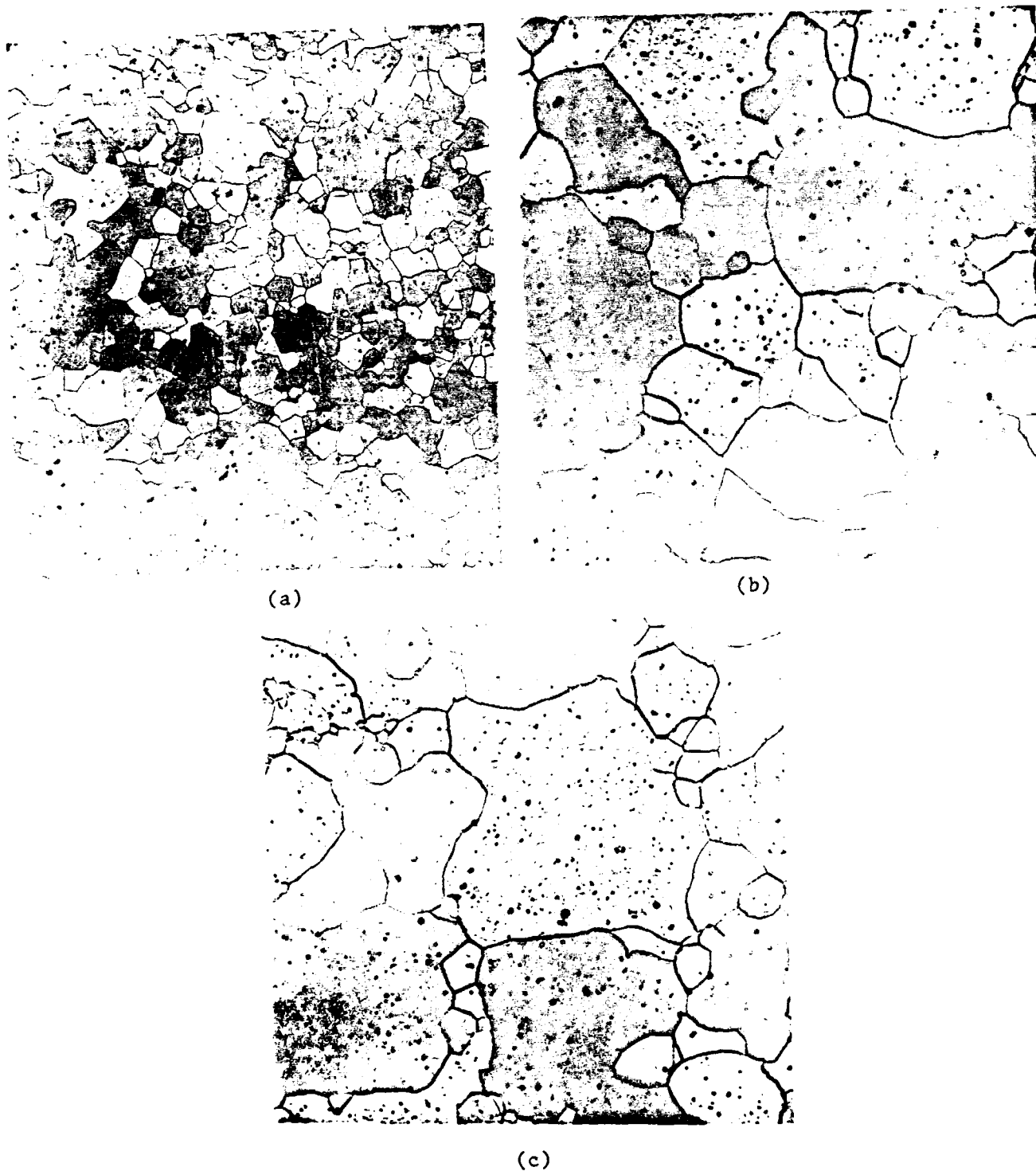
Figure 7. Mo-0.1% Co co-reduced powder densified by the HIP process at 30 Ksi for three hours at the temperatures indicated: (a) 1300°C, (b) 1400°C, and (c) 1500°C, polished and etched microstructures (500X).

31
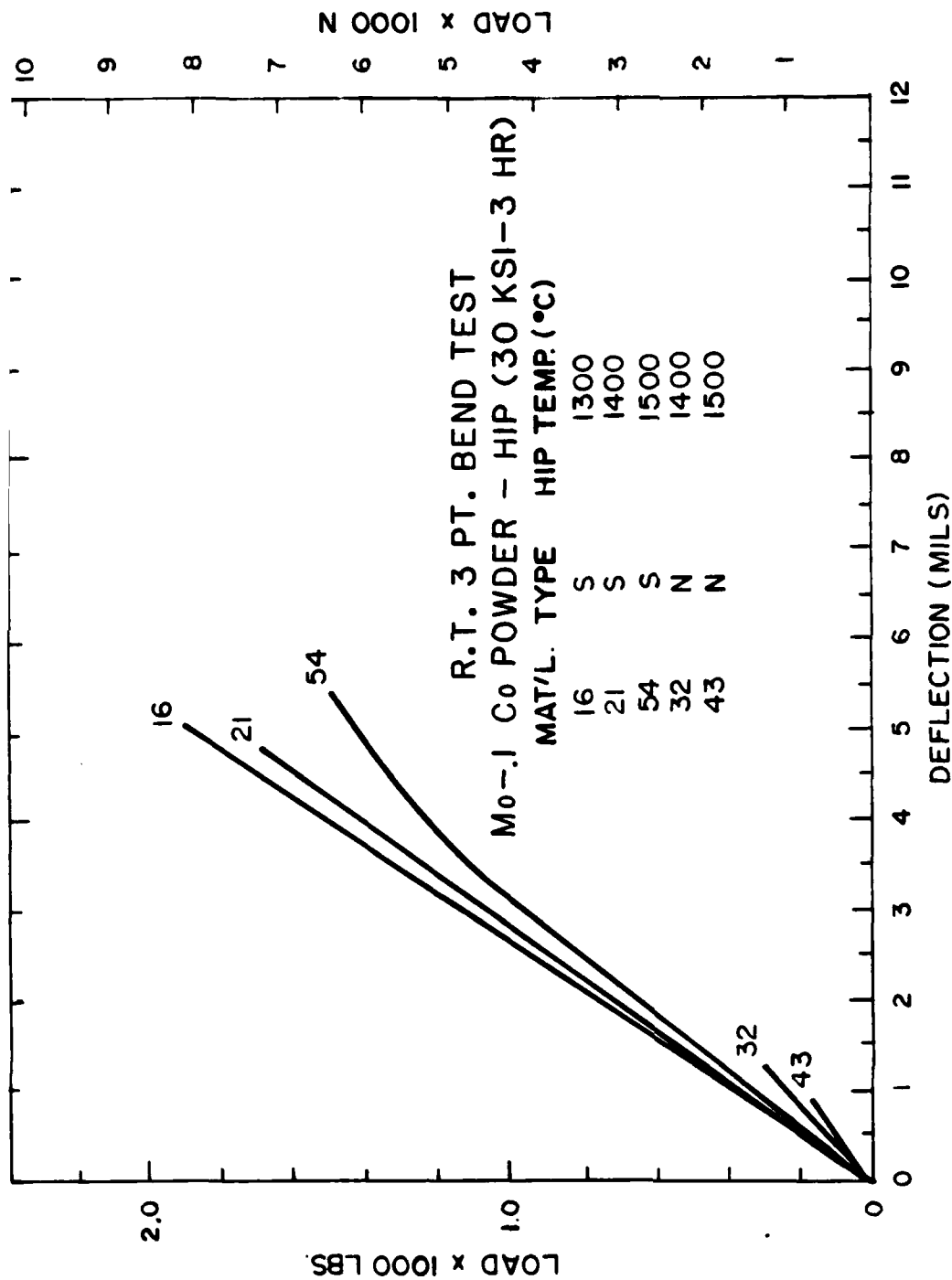
Figure 21. Bending load vs. deflection for Mo-0.1% Co, HIP densified: 30 Ksi, three hours at 1300°C, 1400°C, and 1500°C.

R.T. COMPRESSION TEST
TZM SPHERICAL POWDERS-HIP (30 KSI - 3 HR.)

MAT'L.		HIP TEMP. (°C)
PREP	11	1500
(74 μm)	12	1600
SX-207	16	1500
(24.7 μm)	18	1600

STRAIN x .1%

STRESS x 10 KSI
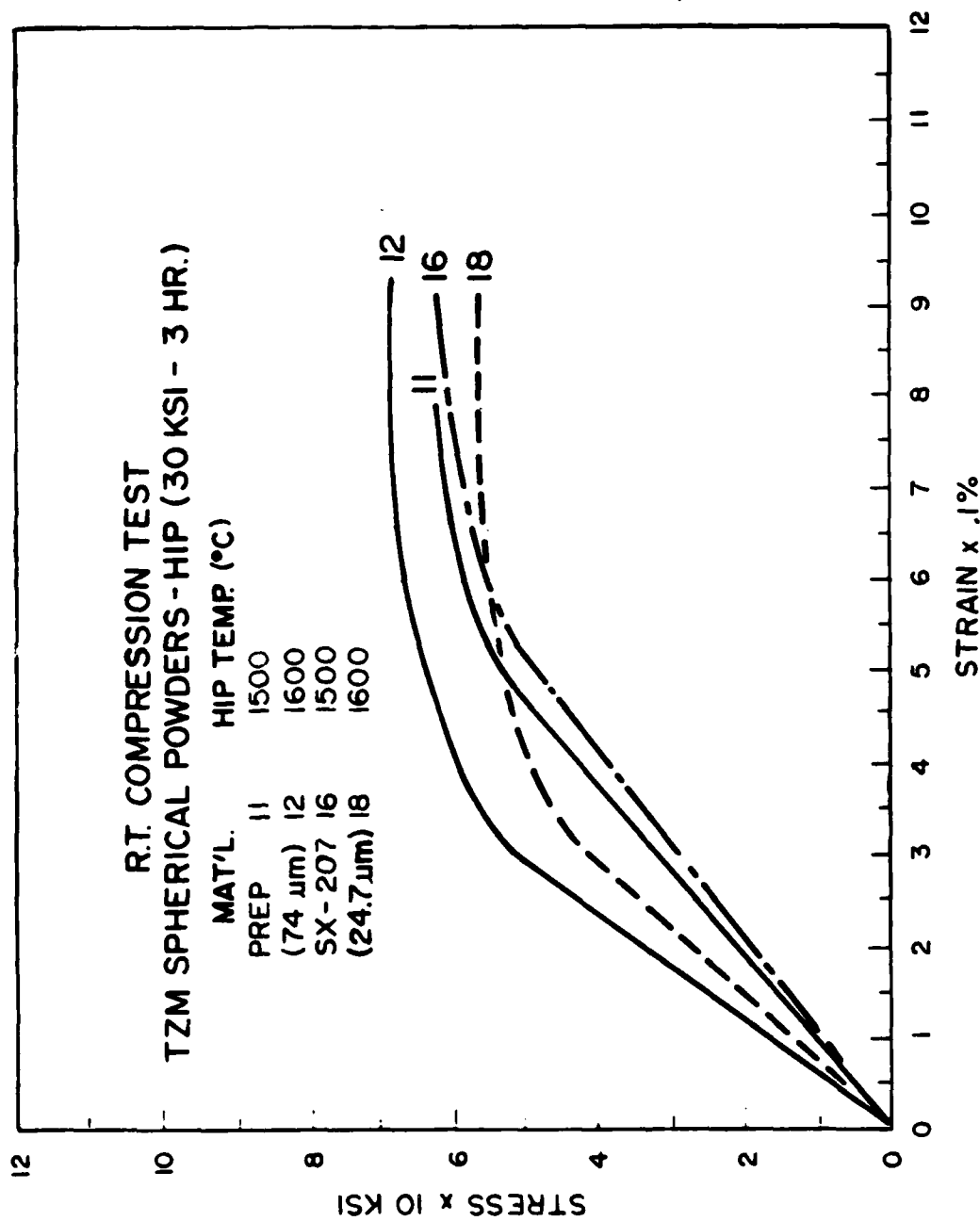
Figure 20.   Compression stress vs. strain for PREP, PMRS, (SX-207) spherical powders, HIP densified:
30 Ksi, three hours at 1500°C and 1600°C.

44
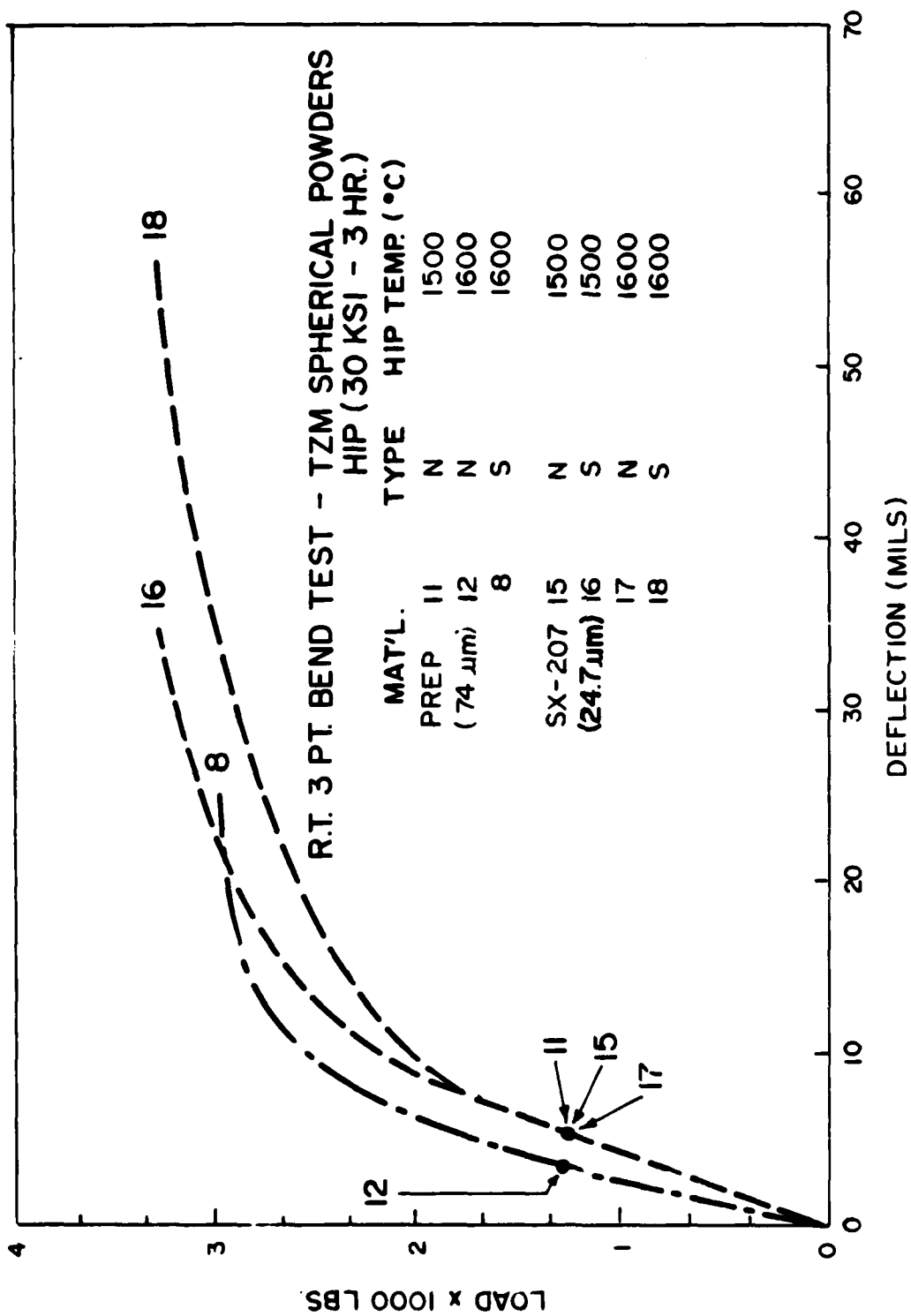
Figure 19. Bending load vs. deflection for PREP, PMRS, (SX-207) spherical powders, HIP densified: 30 Ksi, three hours at 1500°C and 1600°C.
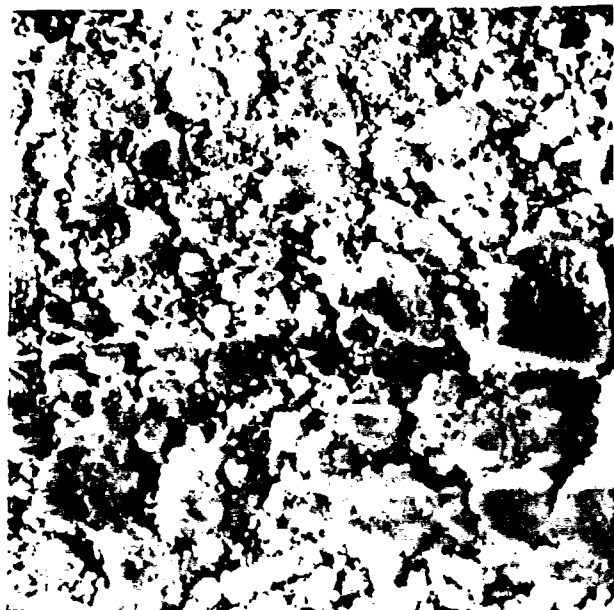
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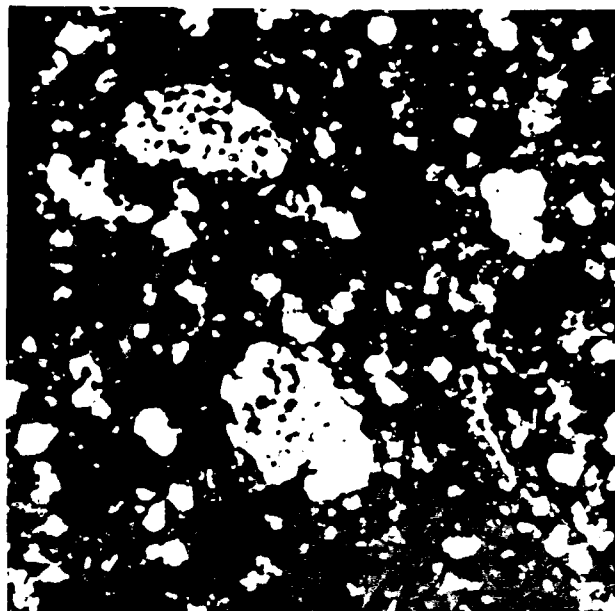
(b)
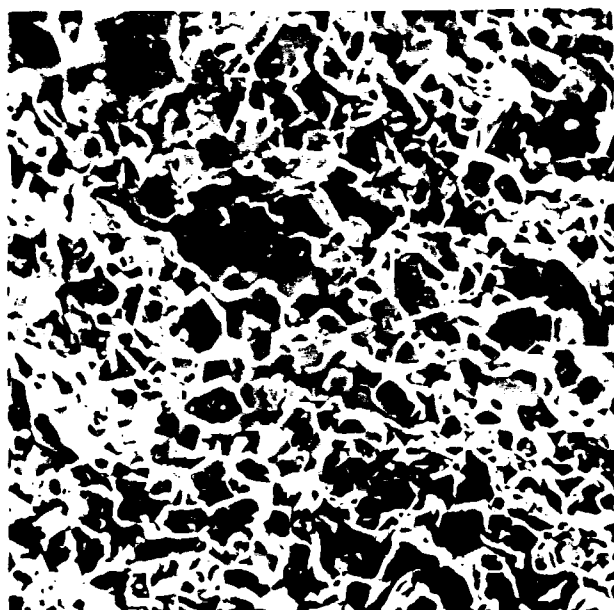
(c)
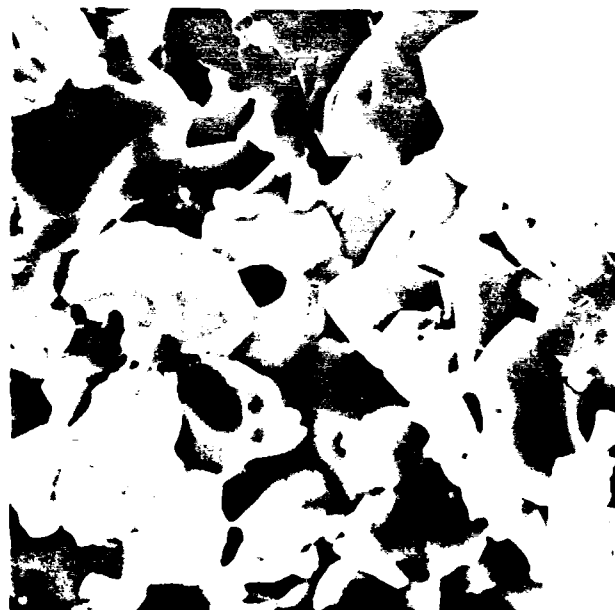
(d)

Figure 18. Alumina (60 wt. %) and molybdenum (40 wt. %) powder – (a) before
HIP densification, SEM (700X) and (b) after HIP compaction at
1500°C, 30 Ksi, for three hours, etched (500X); (c) and (d)
fracture surfaces reveal low energy failure has occurred, SEM
(1000X) and (3000X).

42
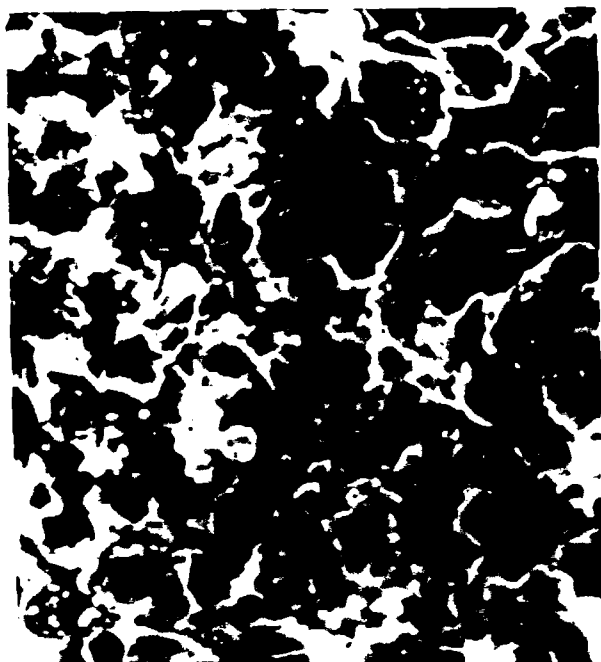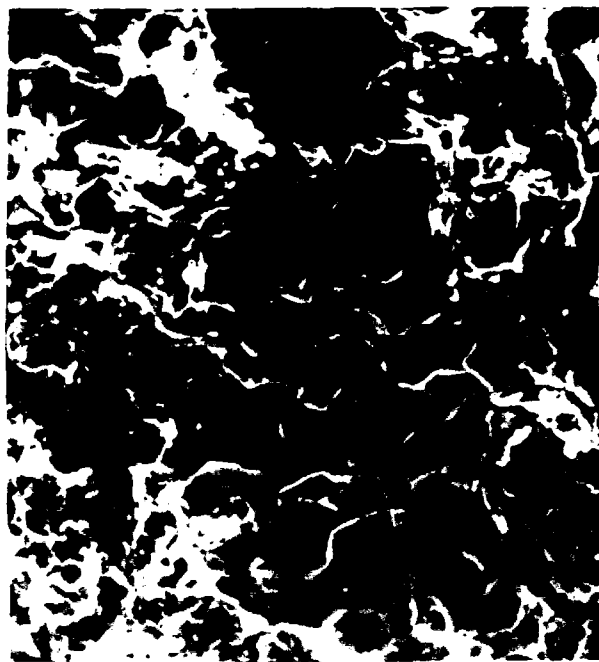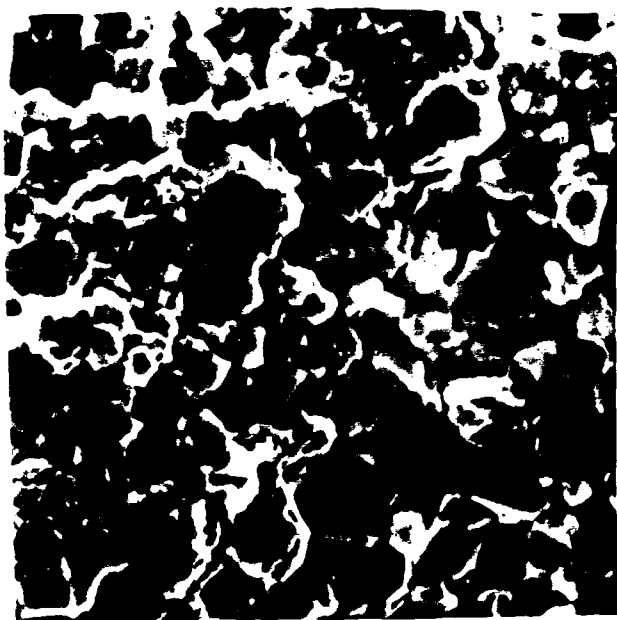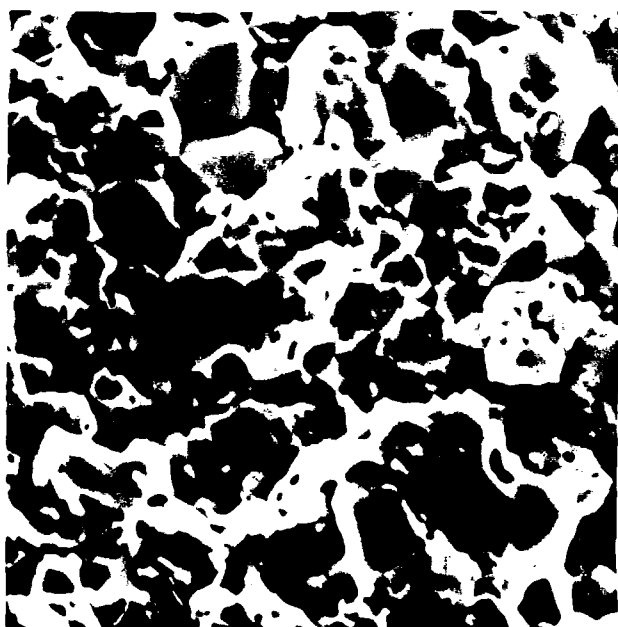
Figure 17. Fracture surface of alumina (5 wt. %) molybdenum (95 wt. %) from compacts HIP densified at 30 Ksi for three hours at temperatures indicated, SEM (5000X). (a) 1400°C, (b) 1400°C, (c) 1500°C, and (d) 1600°C.

41
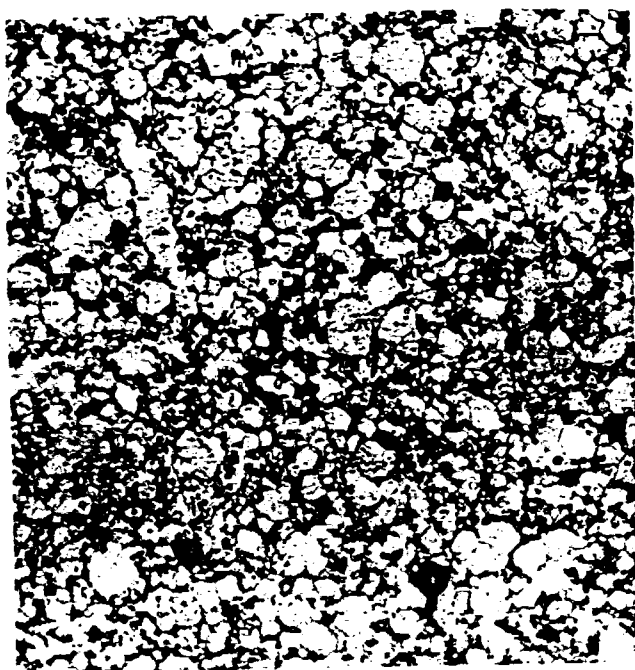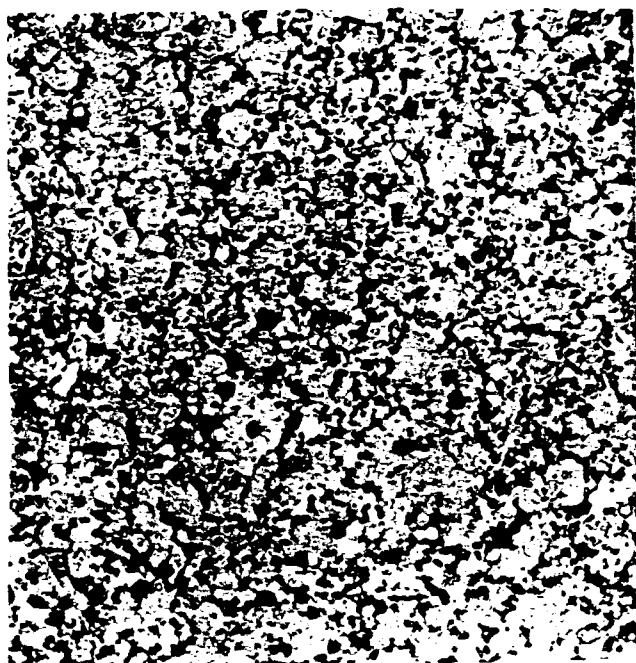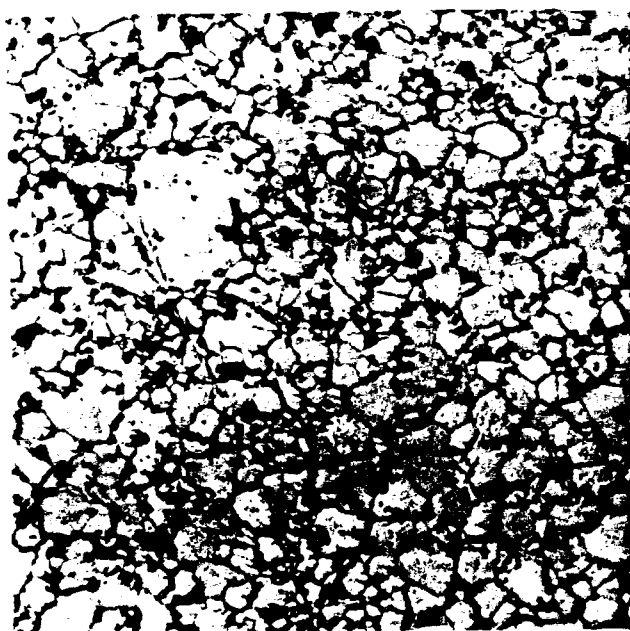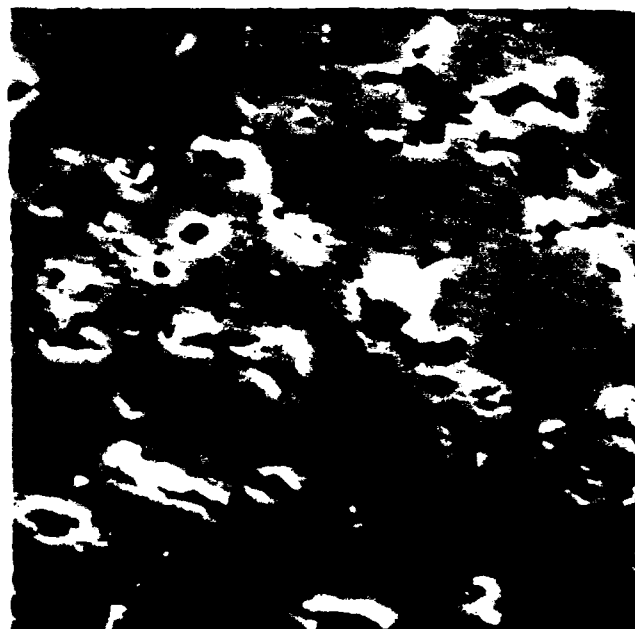
Figure 16. Alumina (5 wt. %) - molybdenum (95 wt. %) mixture HIP densified at 30 Ksi for three hours at temperatures indicated: (a) 1400°C, (b) 1500°C, (c) 1600°C, etched (1000X), and (d) 1500°C, SEM (7000X).
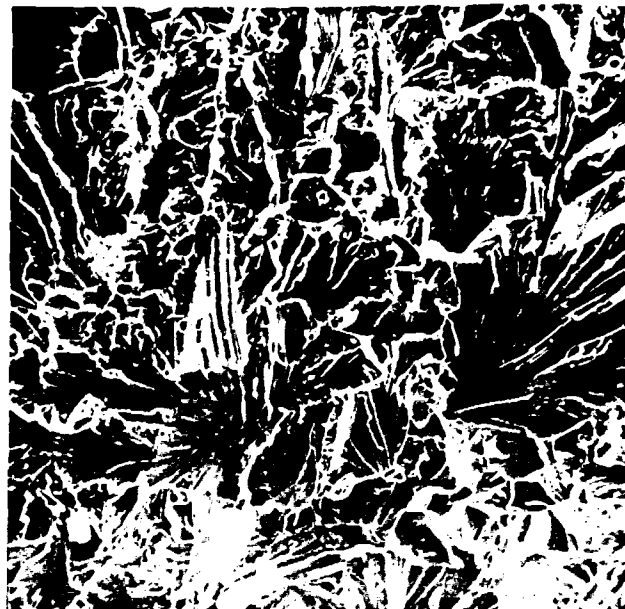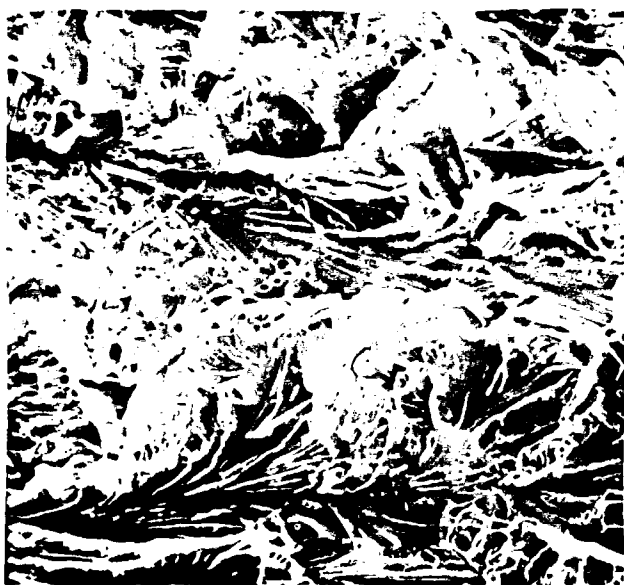
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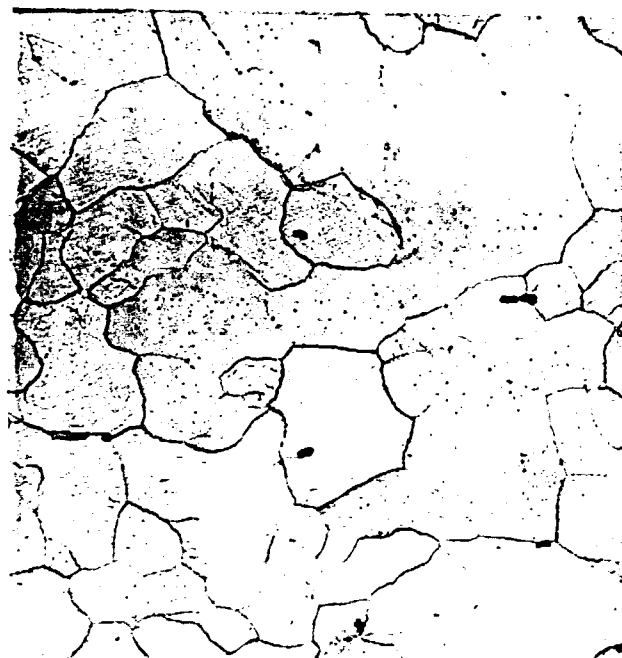
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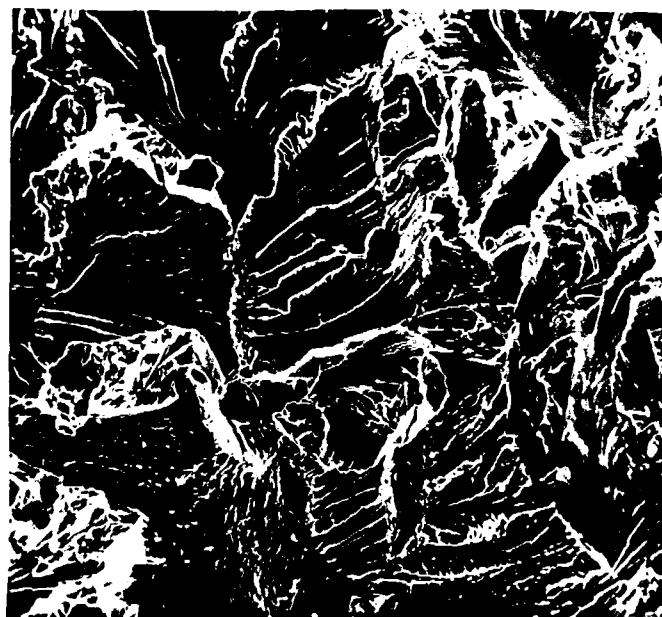
(c)

Figure 15. Microstructures of TZM (P) arc-cast commercially processed
bar stock (4-inch diameter). (a) Longitudinal, etched (500X);
(b) transverse, etched (500X); and (c) fracture surface,
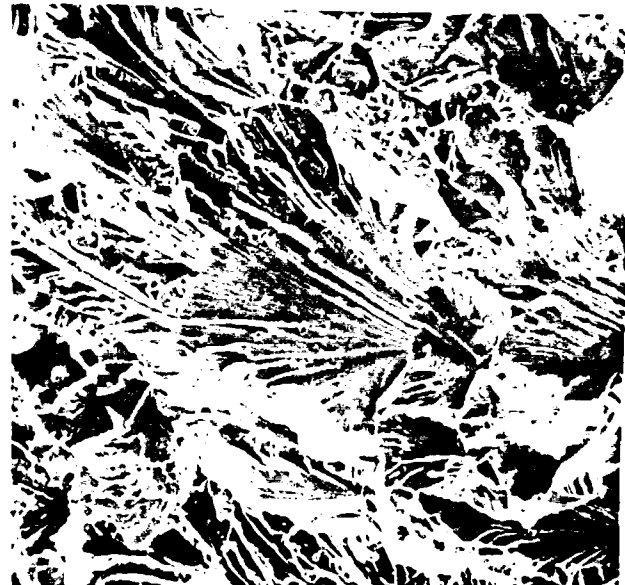notched bar, SEM (500X).

39
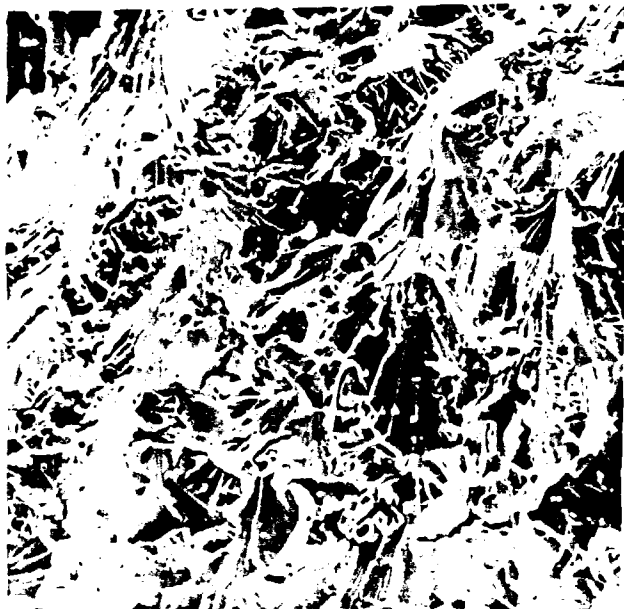
(a)   (b)
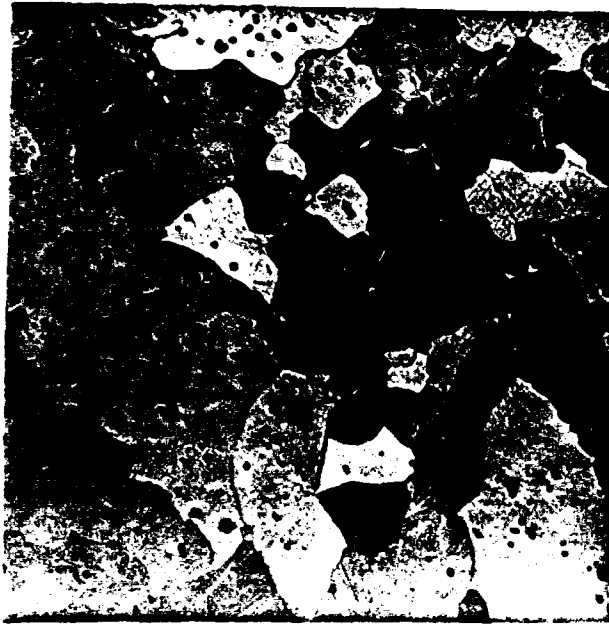
(c)   (d)

Figure 14. Fracture surfaces produced by three-point bending from notched
            specimens cut from 1-1/4-inch diameter bar stock of TZM (GTE)
            after exposure to HIP cycles as indicated: (a) 1400°C, 1.5
            hours, 30 Ksi; (b) 1500°C, 3 hours, 30 Ksi; (c) 1600°C, 3 hours,
            15 Ksi; and (d) 1600°C, 3 hours, 30 Ksi, SEM (500X).
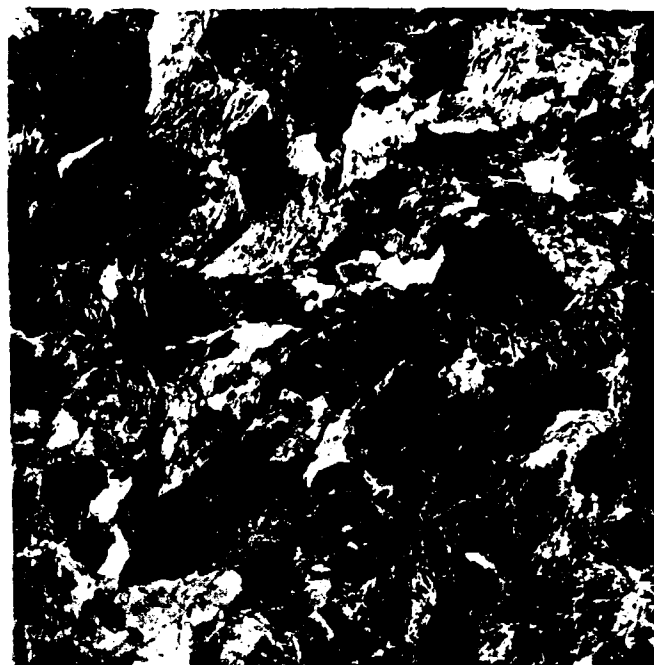
38

Figure 13. Microstructures of pressed and sintered bar stock of TZM (GTE) subjected to HIP processing at temperatures and times indicated: (a) 1400°C, 1.5 hours, 15 Ksi; (b) 1500°C, 3 hours, 30 Ksi; (c) 1600°C, 1.5 hours, 15 Ksi; and (d) 1600°C, 3 hours, 15 Ksi, etched (500X).
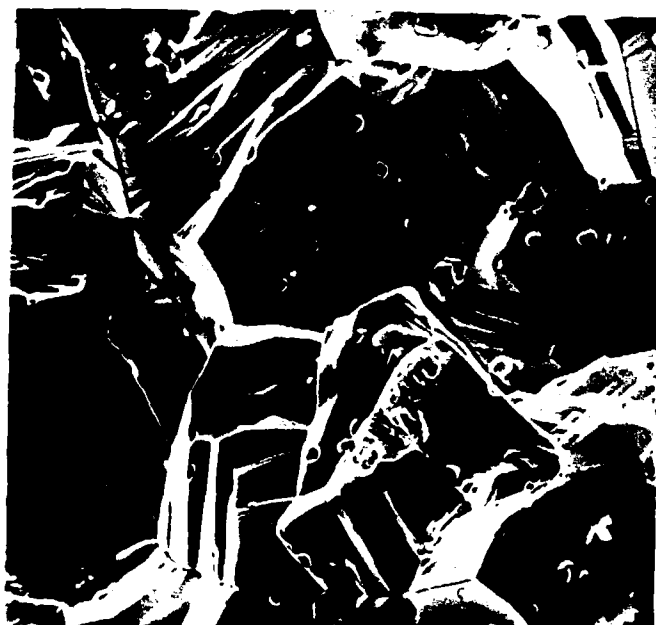
(a)

(b)

Figure 12. Pressed and sintered microstructures of TZM (GTE) bar stock
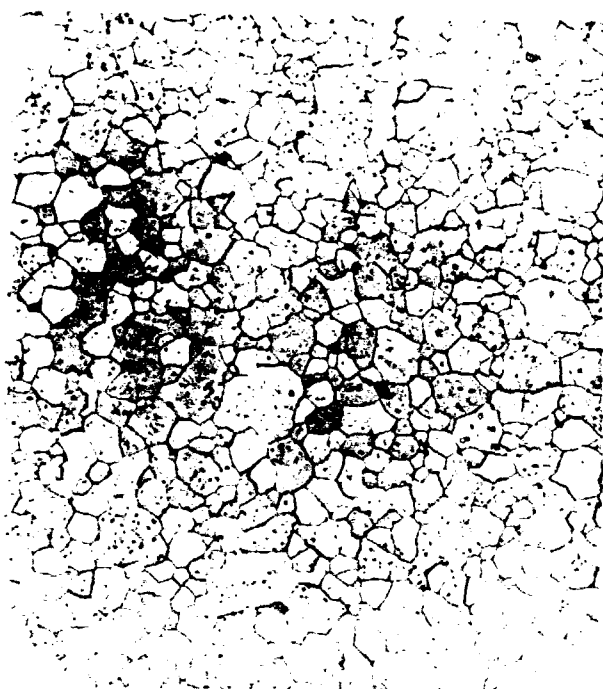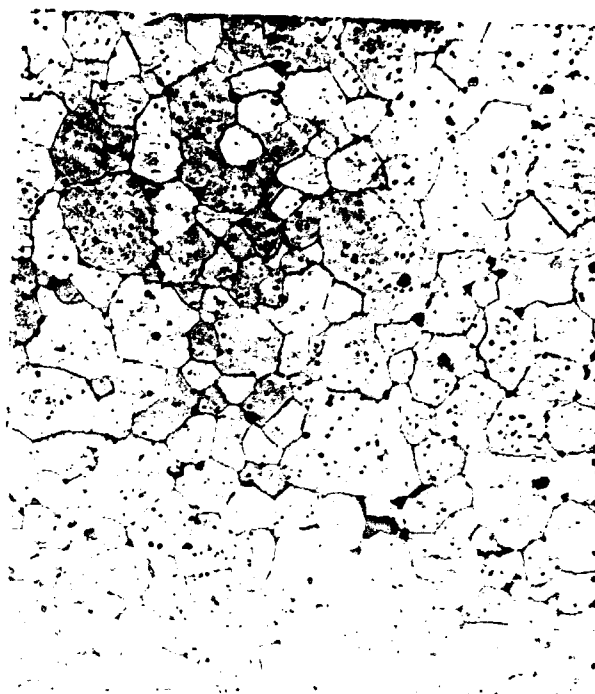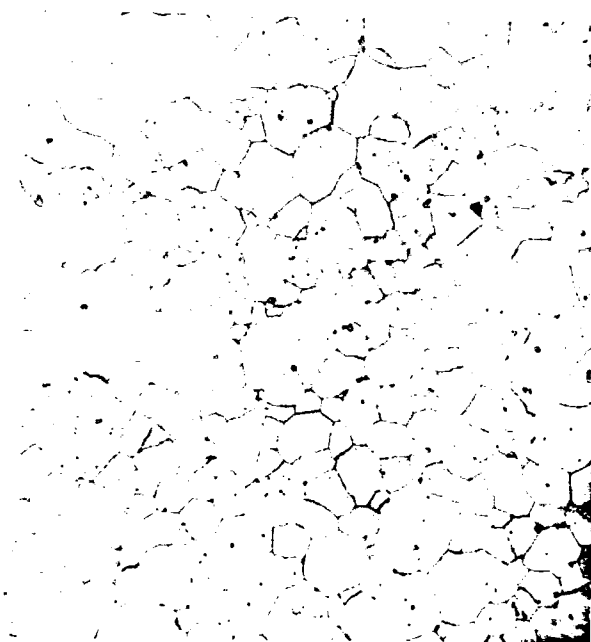not subjected to HIP cycles - (a) 250X and (b) 500X, etched.

36

(a)

(b)

Figure 11. Fracture surfaces produced by three-point bending of smooth bars
(0.394 in. sq.) from molybdenum hydrogen reduced powders
densified by the HIP process at 1500°C and 30 Ksi for three
hours. (a) Fracture using 2 μm powder, SEM (1500X) and (b)
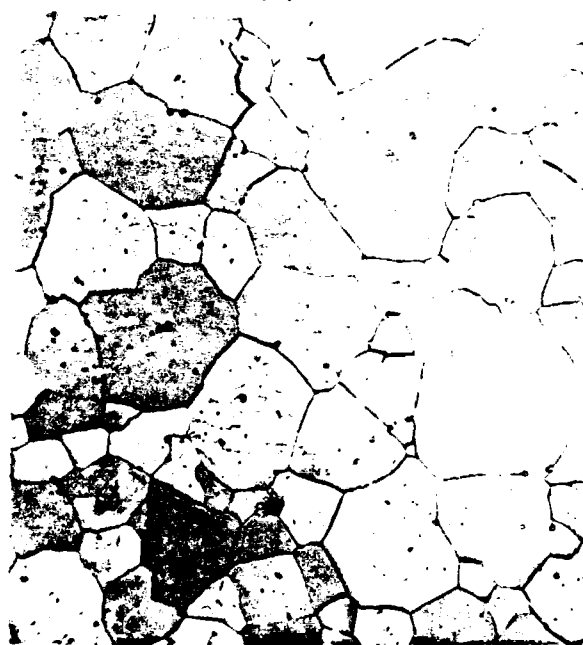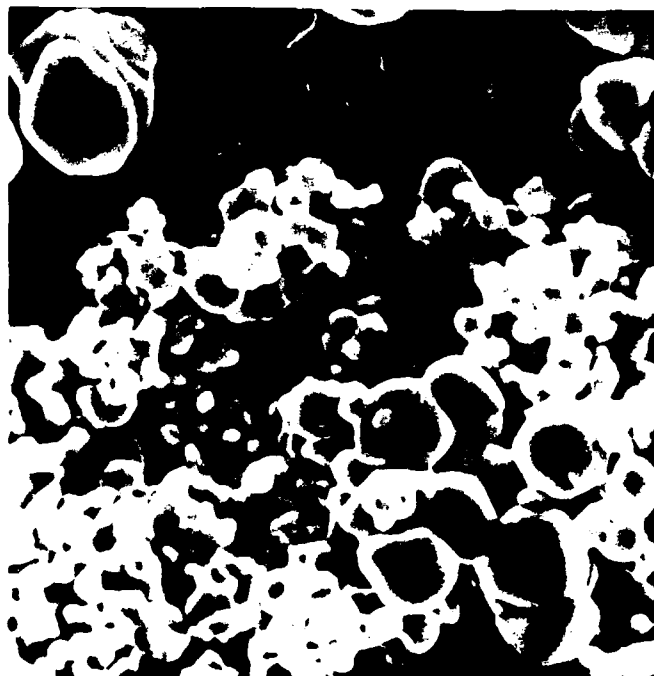fracture using 5 μm powder showing cleavage planes, SEM (1500X).

35

(a)

(b)

(c)

(d)

Figure 10. Two molybdenum hydrogen reduced powders densified by the HIP process at 30 Ksi for three hours at temperatures indicated. 2 μm particle size: (a) 1400°C and (b) 1500°C; 5 μm particle size: (c) 1400°C and (d) 150C°C, etched (500X).
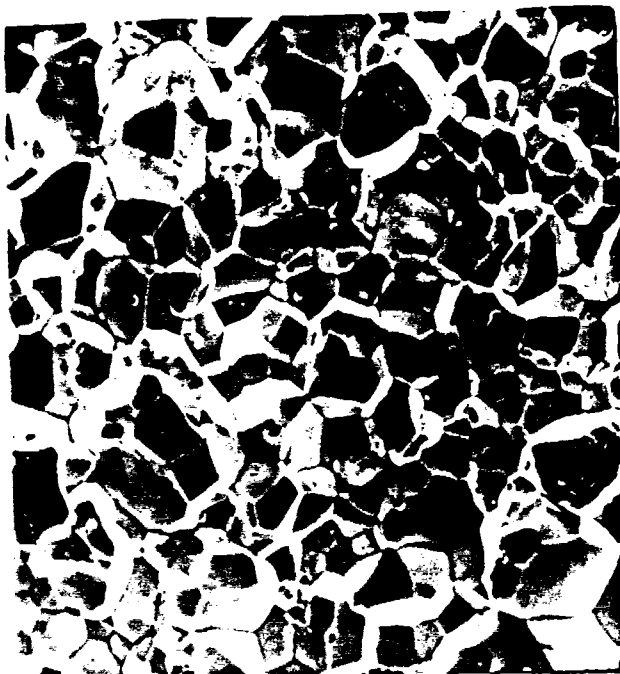
34

(a)

(b)

Figure 9. Molybdenum hydrogen reduced metal powder as-received. (a) 2 µm average particle size (7000X) and (b) 5 µm average particle size (4000X).

(a)
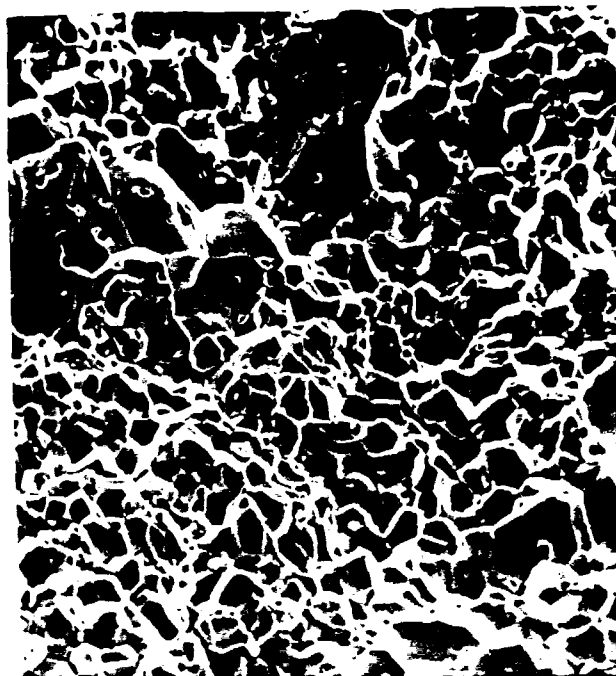
(b)
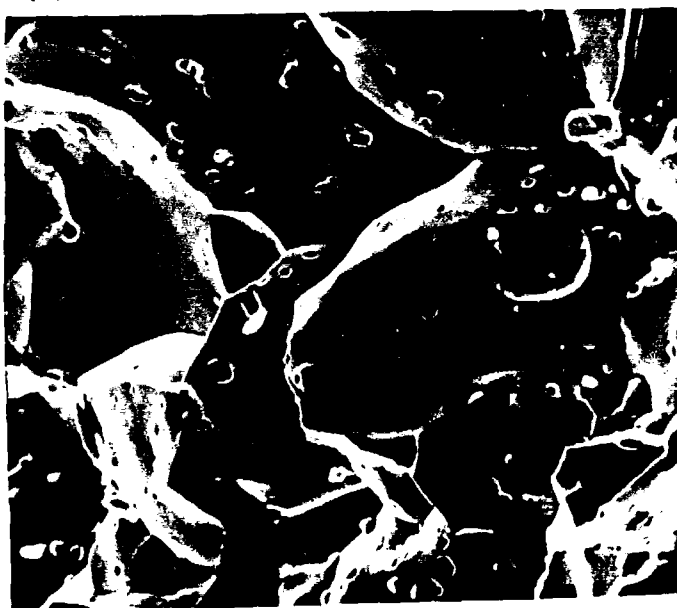
(c)

Figure 8. Intergranular fracture surfaces produced by three-point bending of
bars (0.394 in. sq.) from Mo-0.1% Co powder HIP densified at 30 Ksi
for three hours at temperatures indicated - (a) 1300°C, (b) 1400°C
showing stratification of grain sizes, (c) 1500°C with rounding of
faceted surfaces of large grains, SEM (1500X).
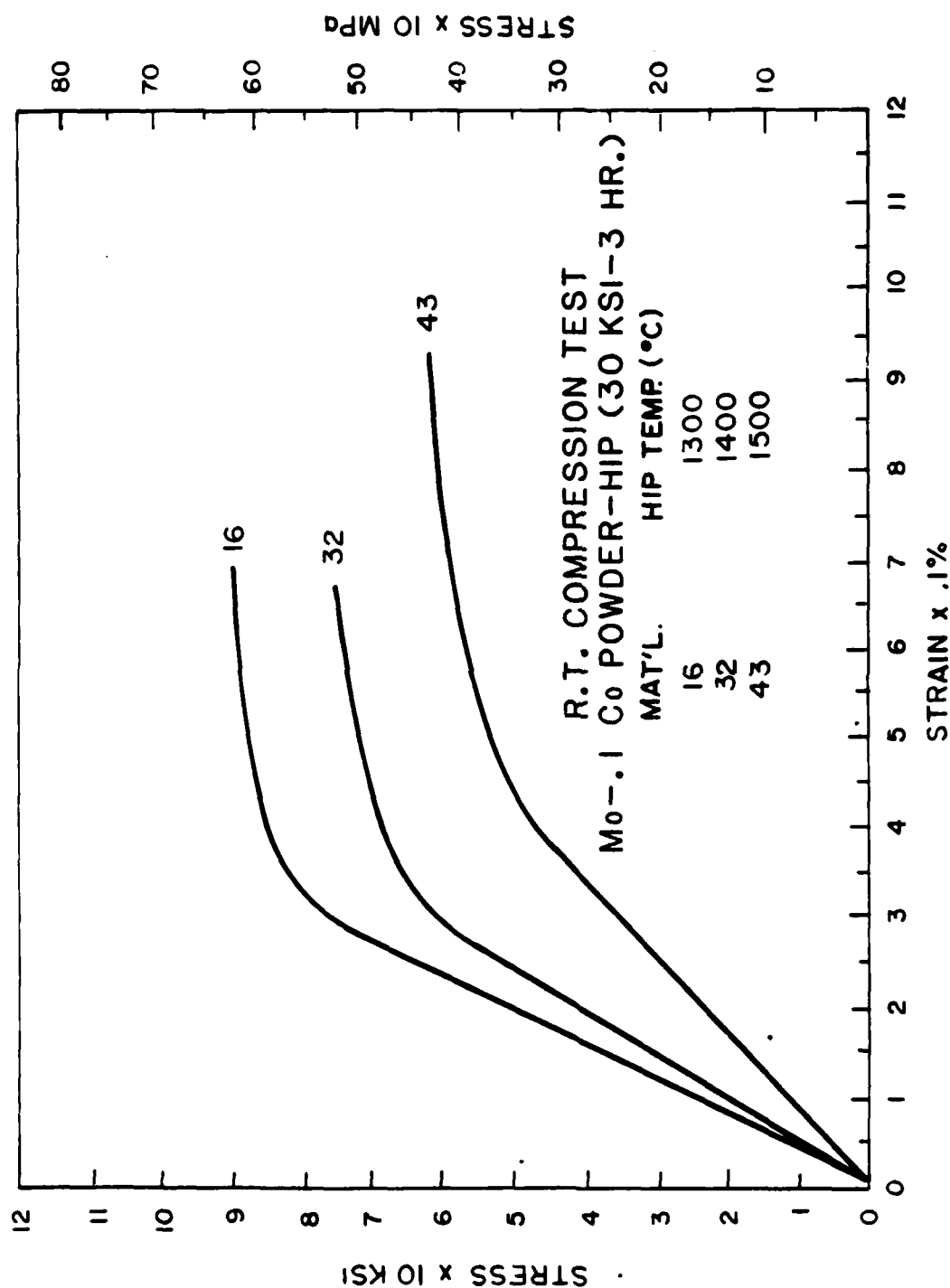
Figure 22. Compression stress vs. strain for Mo-0.1% Co, HIP densified: 30 Ksi, three hours at 1300°C, 1400°C, and 1500°C.

46
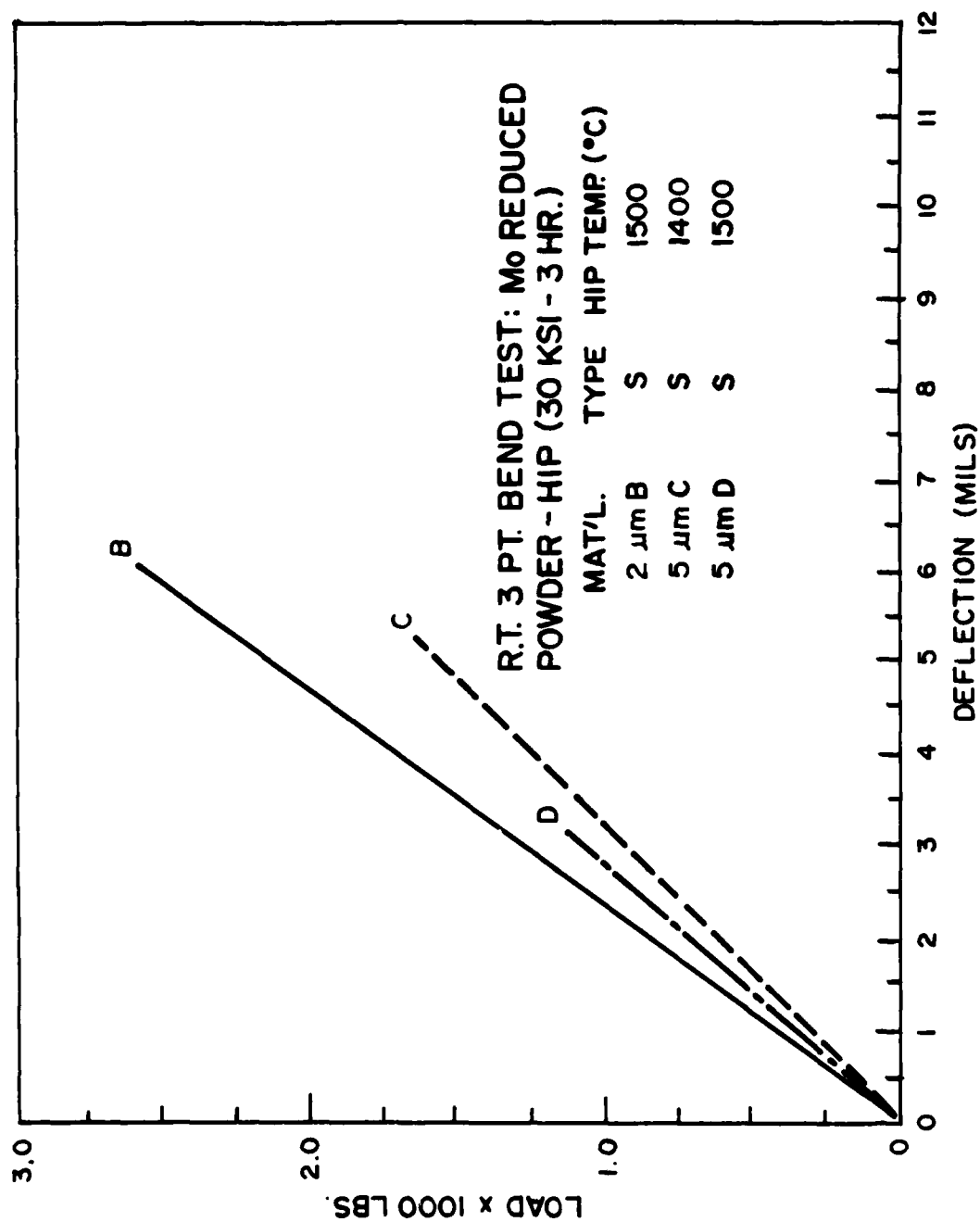
Figure 23. Bending load vs. deflection for 2 μm, 5 μm, molybdenum reduced powders HIP densified: 30 Ksi, three hours at 1400°C and 1500°C.

47
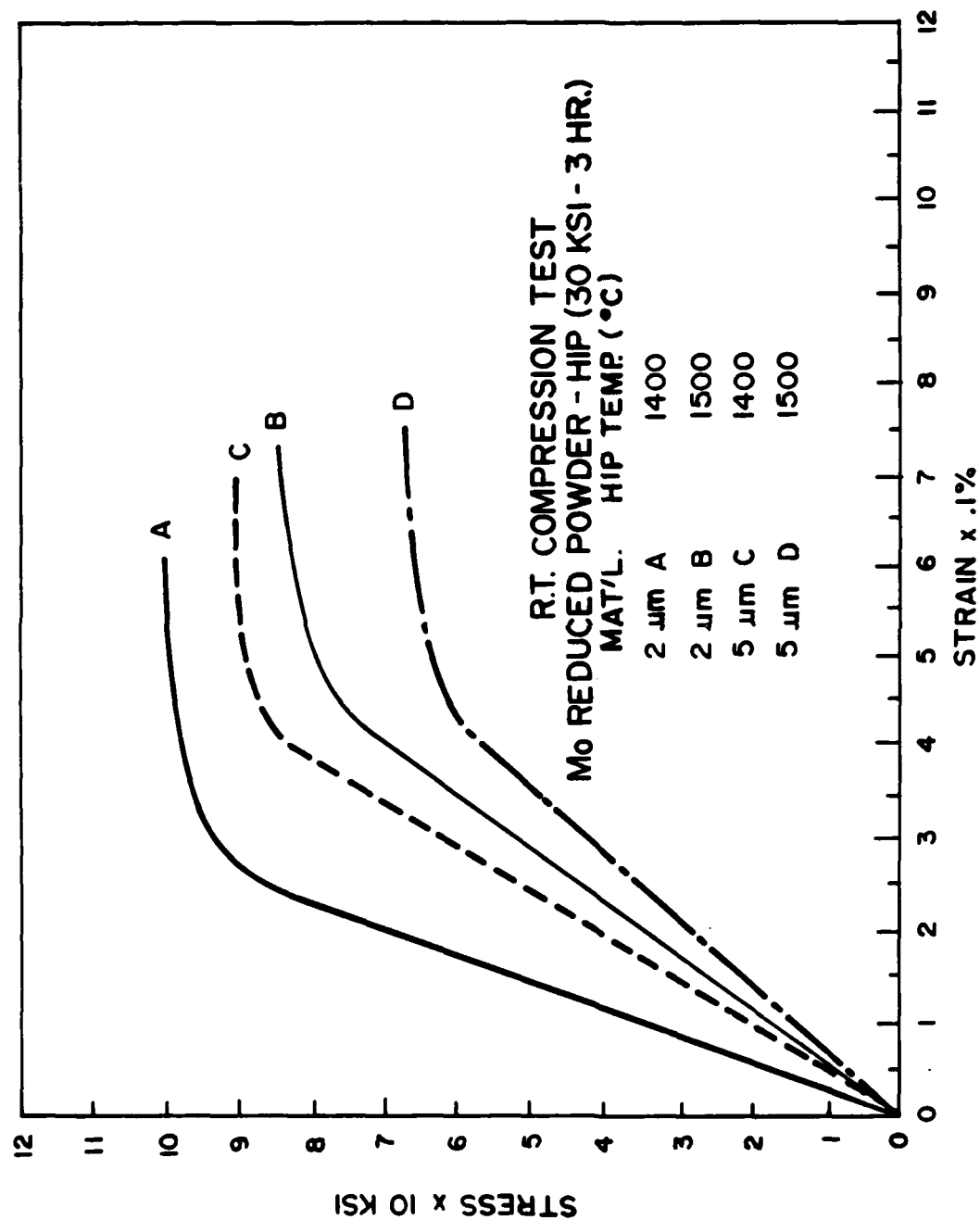
Figure 24. Compression stress vs. strain for 2 μm, 5 μm, molybdenum reduced powders, HIP densified: 30 Ksi, three hours at 1400°C and 1500°C.

48
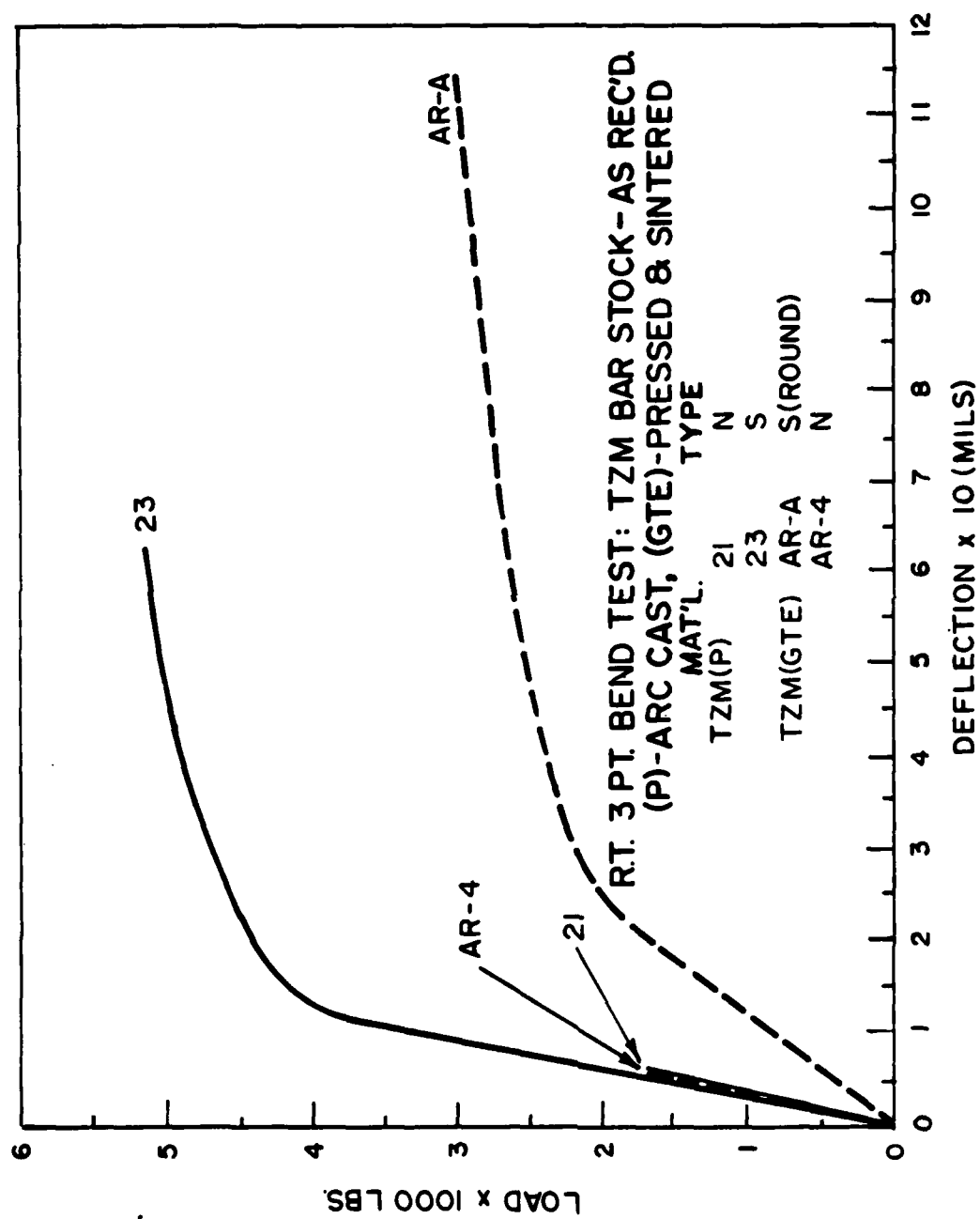
Figure 25. Bending load vs. deflection for TZM (P) arc-cast and TZM (GTE) pressed and sintered bar stock.
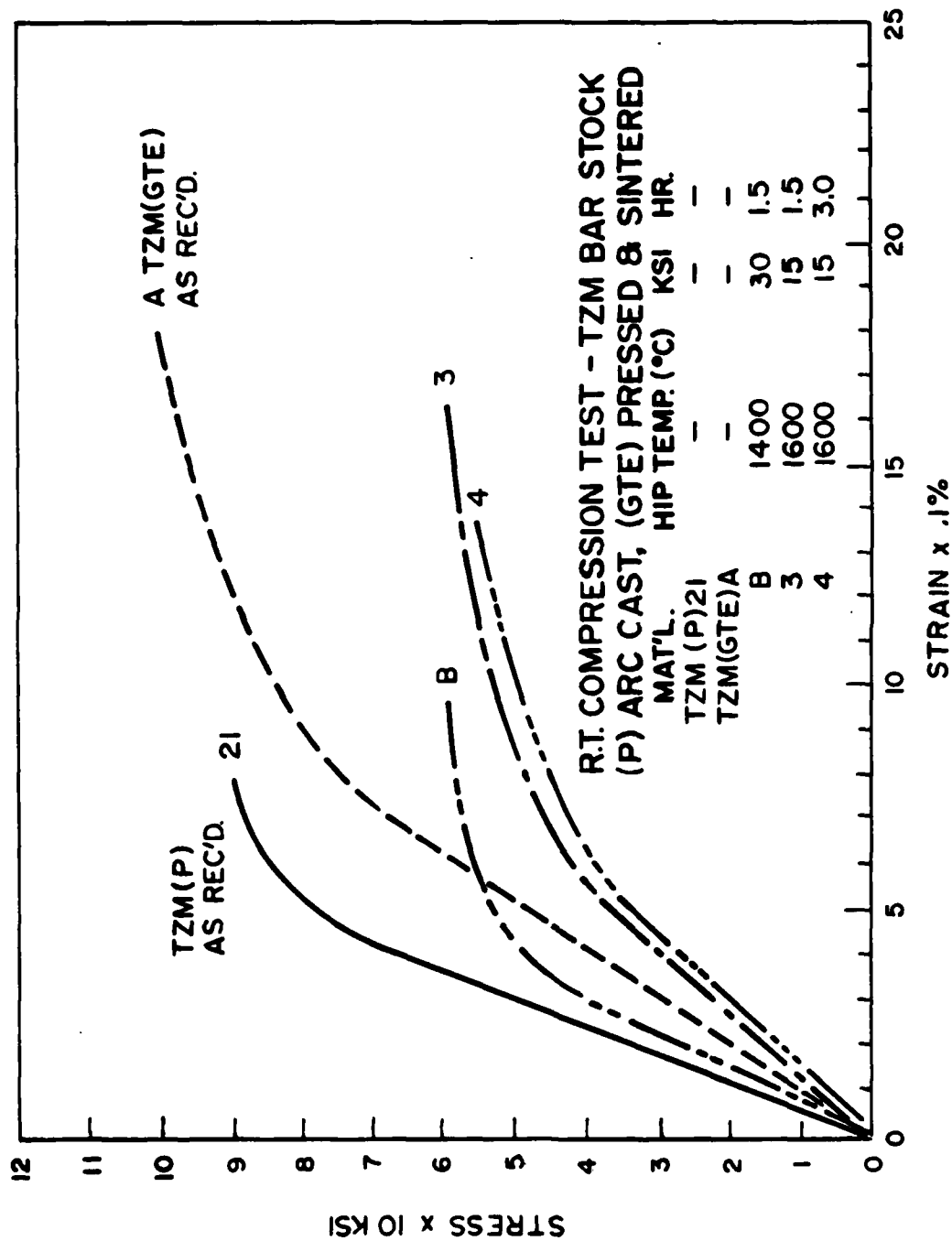
Figure 26. Compression stress vs. strain for TZM (P) arc-cast, as-received condition, and TZM (GTE) pressed and sintered bar stock HIP densified: 15/30 Ksi, 1.5/3 hours at 1400°C and 1600°C.
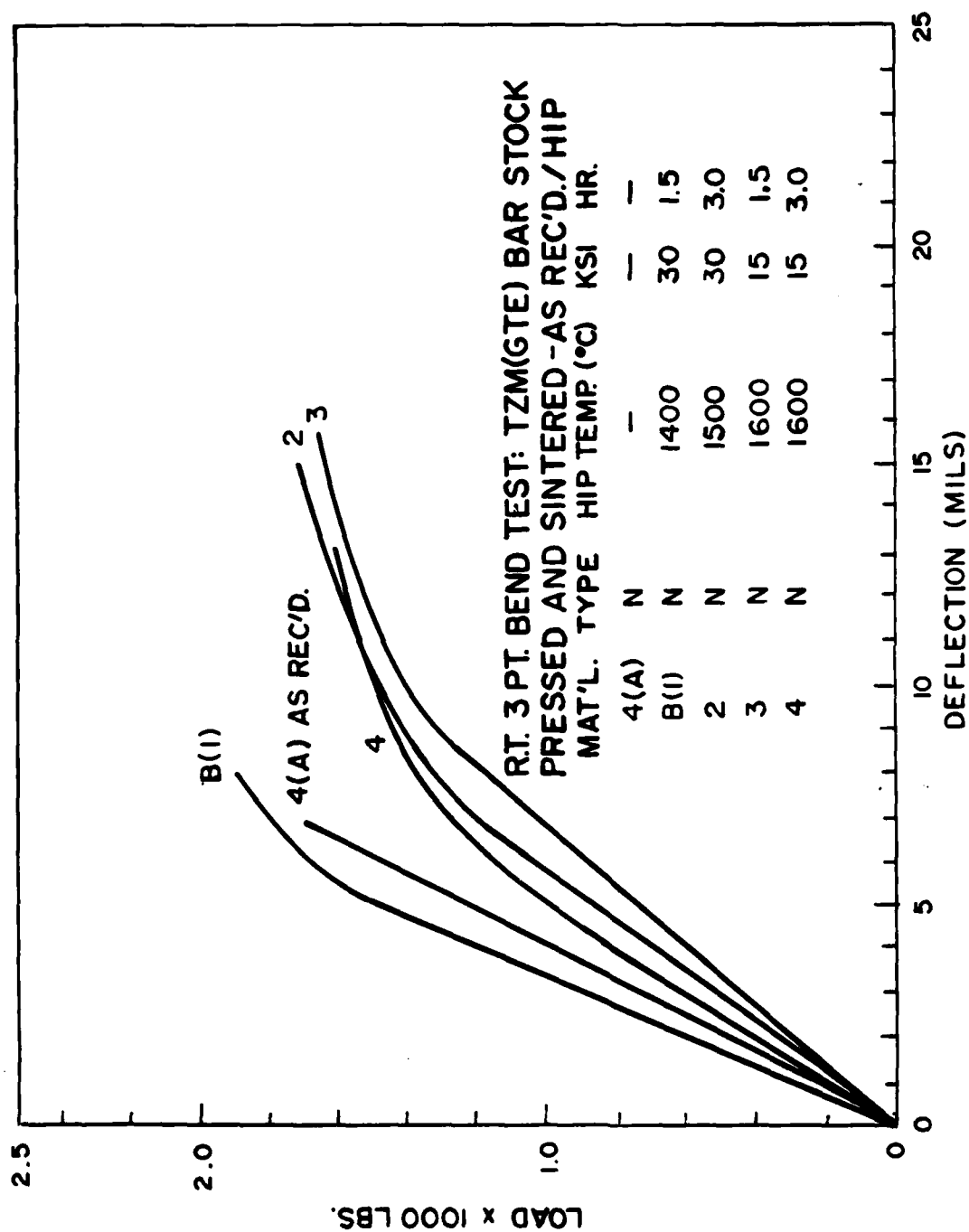
Figure 27. Bending load vs. deflection for TZM (GTE) pressed and sintered bar stock for as-received condition and HIP densified: 15/30 Ksi, 1.5/3 hours at 1400°C, 1500°C, and 1600°C.

51
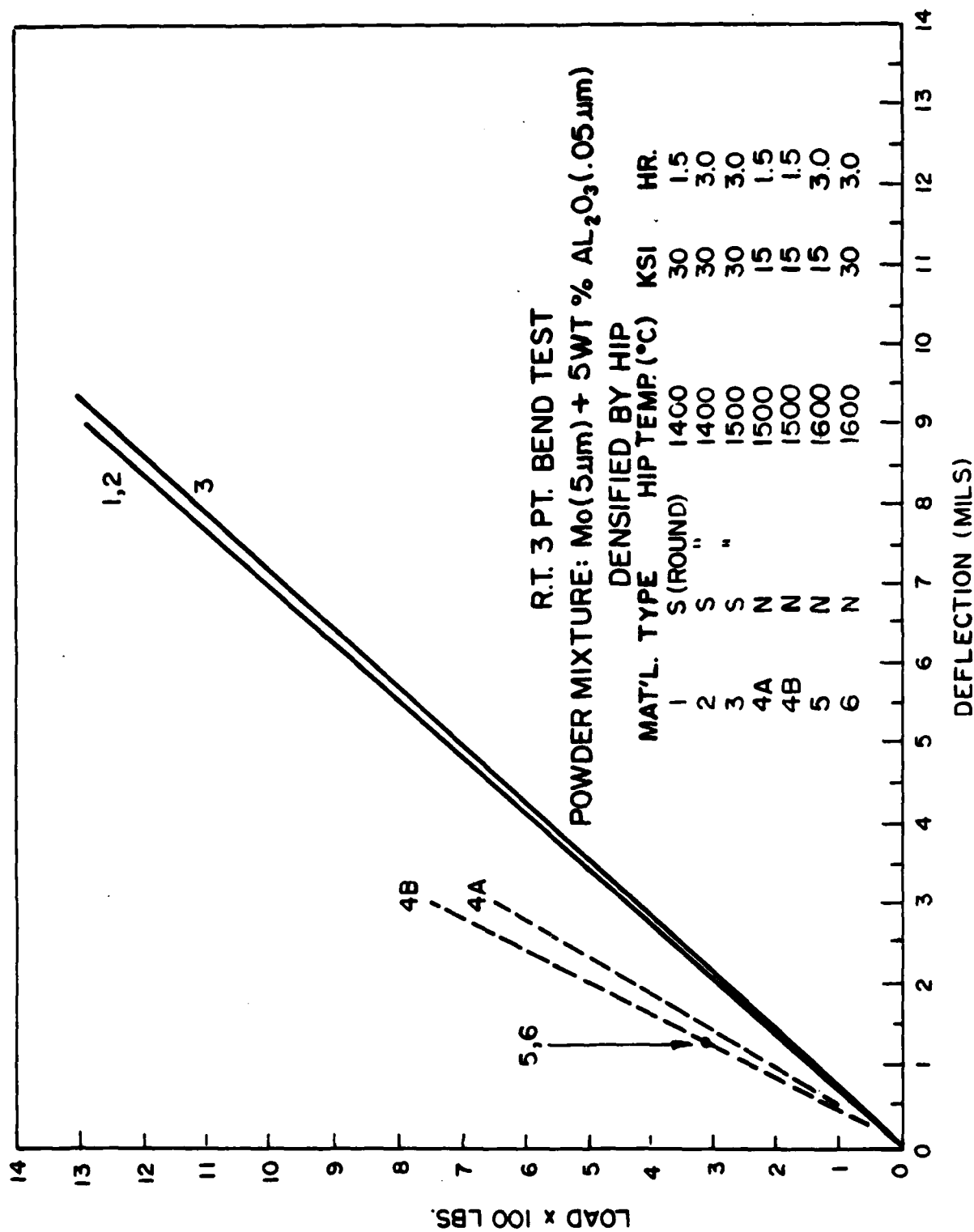
Figure 28. Bending load vs. deflection for Mo-5Al₂O₃ (wt. %) HIP densified: 15/30 Ksi, 1.5/3 hours at 1400°C, 1500°C, and 1600°C.

R.T. 3 PT. BEND TEST

TZM: ARC CAST (P) AND HIP SPHERICAL POWDER (SX-207)

MAT'L.		TYPE	HIP TEMP. (°C)	KSI	HR.
(SX-207)	15	N	1500	30	3
	16	S	1500	30	3
	17	N	1600	30	3
	18	S	1600	30	3
ARC CAST (P)	21	N	—	—	1
	22/23	S	—	—	1

DEFLECTION (MILS)

LOAD x 1000 LBS.

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Figure 29. Bending load vs. deflection for TZM (P) arc-cast, as-received condition, and TZM (PMRS) SX-207 spherical powder HIP densified: 30 Ksi, three hours at 1500°C and 1600°C.
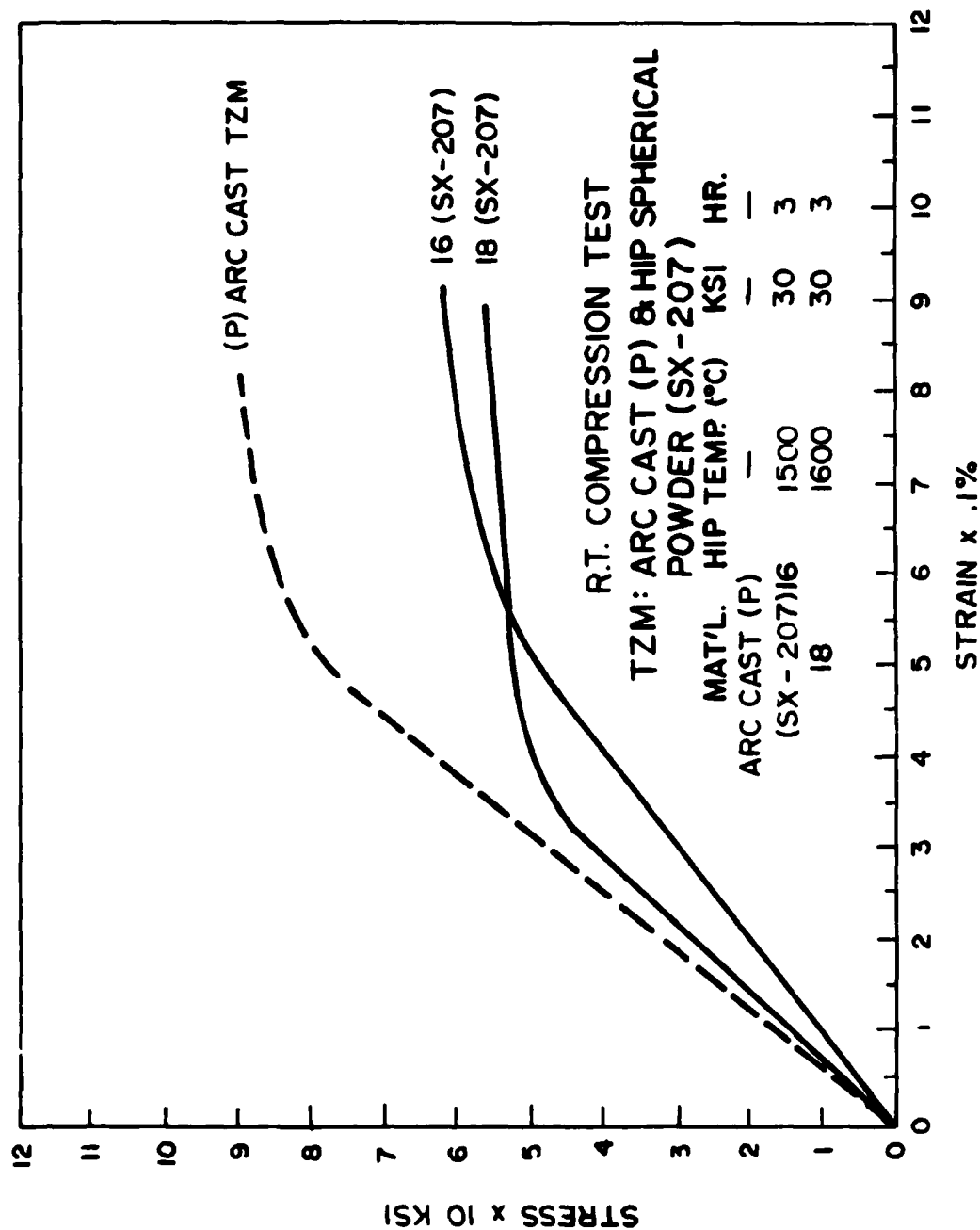
Figure 30. Compression stress vs. strain for TZM (P) arc-cast, as-received condition bar stock and TZM (PMRS) SX-207 spherical powder HIP densified: 30 Ksi, three hours at 1500°C and 1600°C.

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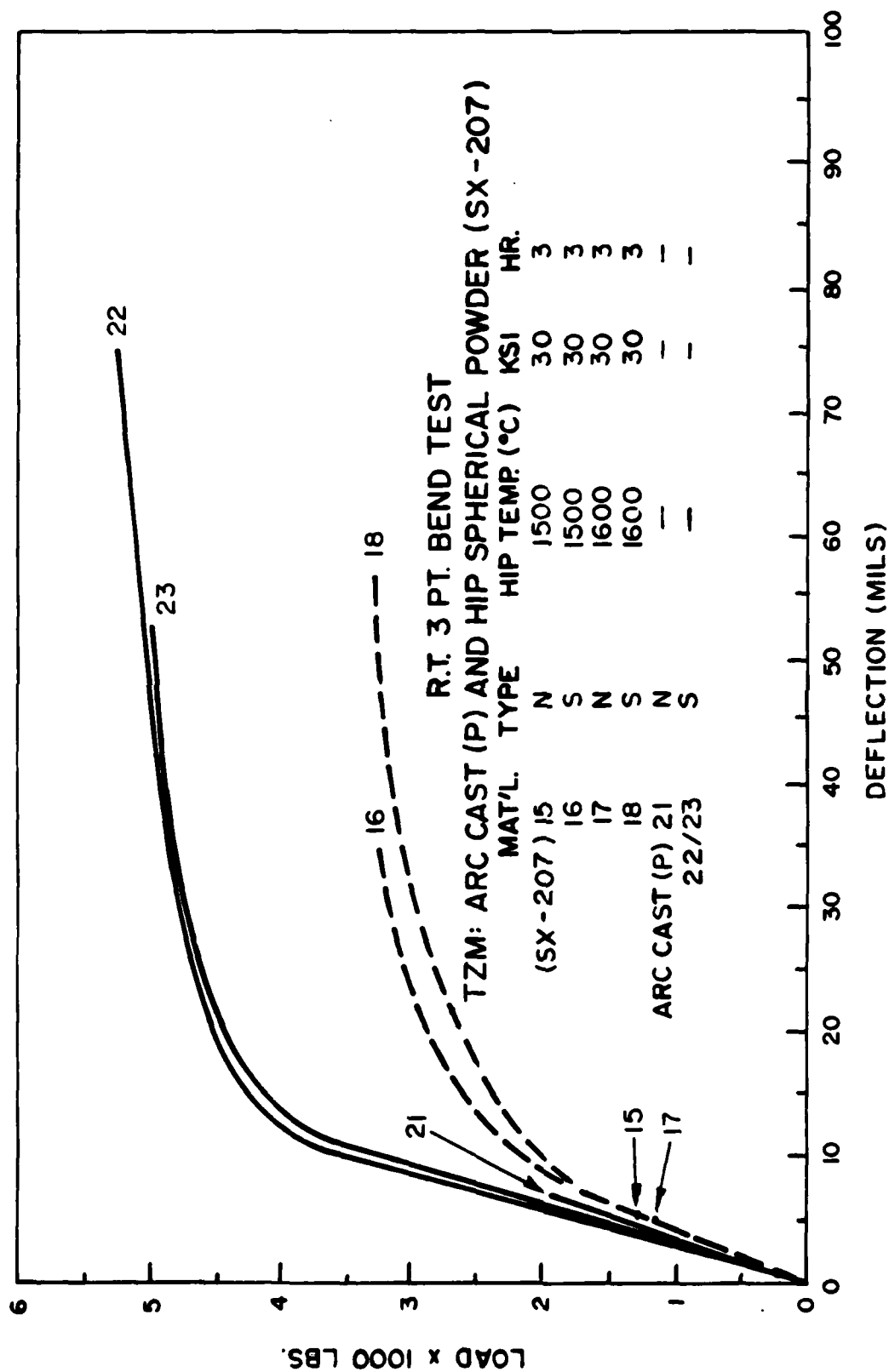|	NO. OF COPIES
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