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RECENT ADVANCES IN HIGH DENSITY TUNGSTEN COMPOSITE PROCESSING

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Conventional liquid phase sintering has been the mainstay for processing tungsten alloy composites for the past several decades. New application demands being placed on these high density composites have resulted in the emergence of new processing approaches aimed at developing a new generation of high density tungsten composite materials. Recent investigative studies into rapid solidification technology, solid state sintering, mechanical alloying, and explosive compaction are reported here to highlight these recent processing trends.

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INTRODUCTION

Traditional processing to obtain high density tungsten (heavy) alloys has usually relied upon liquid phase sintering operations. In these operations, a typical blended mixture of metallic elemental powders of tungsten, iron, and nickel is cold isostatically compacted and then subjected to a high temperature sintering step in a hydrogen reducing gas environment. It is during this high temperature (>1400°C) step that full densification of the compacted blended powders occurs. The resultant particulate composite consists of large tungsten particles embedded in a tungsten-rich matrix alloy of resolidified iron-nickel (fig. 1). Over the years, specific industrial processing procedures have been developed to minimize such detrimental aspects as residual porosity, embrittling impurity segregation, and nonuniform matrix distribution that often can intrude themselves into standard liquid phase sintering operations (ref 1). In spite of these process improvements, the inherent liquid phase sintering process still results in substantial tungsten grain growth to give significantly large tungsten particles, as well as, a nominal degree of tungsten-tungsten particle contacts (i.e., contiguity). It is these factors, even in a carefully processed material, that are often deemed responsible for the undesirable properties these composite materials display under unique high strain rate deformation conditions. Several studies have already substantially demonstrated the influence that the degree of contiguity has on quasi-static fracture in these alloys (ref 2). More recently, the tungsten grain size in these composites has been correlated with shear bandwidths (ref 3). It is hypothesized that it is the timely presence of these shear bands during dynamic compressive failure that characterizes the ballistic penetration capabilities of these materials (ref 4). The tungsten particle size may therefore be an important factor in establishing the ability of these composites to undergo appropriate localized adiabatic shear failure during high strain rate compressive loadings.

The persistent desire for a more refined microstructure and reduced contiguity in these composites has generated a need for new processing approaches aimed at developing a new generation of these materials. Major material processing advances occurring during the past decade in the nonrefractory metals field has provided the impetus for considering the merits of these new material processing operations for tungsten and their adaptability to the tungsten system. What will therefore be discussed respectively in this report are recent investigative studies into the use of rapid solidification processing (RSP), solid state sintering, mechanical alloying, and explosive compaction techniques as a means of developing high density tungsten materials that will have unique microstructures and chemistries. It is believed that these unique materials hold the promise of exhibiting desirable deformation behavior under high strain rate conditions.

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RAPID SOLIDIFICATION TECHNOLOGY

Rapid solidification processing (RSP) of nonrefractory metals has made major strides during the past decade in providing new alloys with enhanced properties (ref 5). The improvements in mechanical properties resulting from microstructural refinements obtained by rapid solidification processing has been demonstrated for aluminum (refs 6 and 7) and steel (refs 8 and 9) alloy systems. The prospects appeared promising for applying this same microstructural refining technology to high tungsten content alloys. An early effort (ref 10) used the traditional RSP technique of ribbon melt spinning and commutation to obtain flaked powders. The powders were subsequently consolidated by a hot extrusion operation to provide the final bulk material. The alloy chosen for study was a W-Ni-Fe-B system with tungsten compositions ranging from 40 to 90%. The boron composition ranged between 0.5 and 1.1 weight percent; the higher boron concentrations being used with the higher tungsten content material in order to enhance the castibility and wettability of the ribbon. The final consolidated microstructure consisted of both tungsten and metallic boride particles(1-5 microns) embedded in a nickel based matrix alloy. The finely resolved microstructure for a 50W-35Ni-14Fe-1B (weight percent) alloy (top) and a 65W-24Ni-10Fe-1B alloy (bottom) is shown in figure 2.

Mechanical properties for the 65W alloy were very good (910 MPa yield strength, 1262 MPa ultimate tensile strength, 13% elongation, and an unnotched impact strength of 209 joules) considering that the microstructual refinement was achieved with a single step solid state consolidation operation. A comparable solid state sintering operation of a traditional 70% tungsten heavy alloy powder mix (W-Ni-Fe) also resulted in a similar grain refinement but much lower strength properties (ref 11). Furthermore, a liquid phase sintering consolidation for these RSP powders would have most likely resulted in substantial tungsten and boride particle growths and thereby a decidedly less ductile material.

In this project, higher density tungsten alloys than 65 weight percent tungsten could not reliably be melt spun due to the excessive wear of the ceramic crucible nozzle in the pilot plant equipment. A variety of ceramic nozzles were tried but all were unsuccessful in maintaining a controlled molten metal flow stream. Considering that the casting temperatures exceeded 2700°C, the durability of most ceramic nozzles would be in question. Noncontacting schemes therefore need to be devised to affect the adaptability of RSP to higher tungsten alloys. Preliminary studies into consumable electrode splat quenching (ref 12) and plasma generated rapidly solidified tungsten alloy powders (ref 13) were undertaken. In the latter technique shown in figure 3, agglomerated powders containing the appropriate constituents are produced by a spray drying operation. These powders are then drawn through a plasma flame where they are rapidly melted and solidified to produce spherical tungsten powders that can be either dispersion or solute solution strengthened. The powders are then used as

feedstock material in various experimental consolidation processes. Pioneer liquid phase sintering studies (ref 14) using these RSP powders revealed such excessive dissolution of the tungsten particles as to negate the benefits of prior plasma processing. More sophisticated consolidation methods for these unique powders therefore need to be explored. However, a note of further caution is advised. Unlike the monocrystalline tungsten powders typically employed in liquid phase sintering operations, these plasma rapidly solidified processed powders have complex microstuctures. The fracture characteristics of a large RSP W-HfC dispersion strengthened particle is shown in figure 4. The particle is multigrained and shows interior porosity. Further work is therefore needed to improve upon the microstructure of these RSP particles before they can be consolidated into bulk forms.

SOLID STATE SINTERING

To avoid the large tungsten grain growth normally arising from liquid phase sintering of tungsten heavy alloys, many have proposed employing solid state sintering as a means of obtaining a tungsten composite consisting of much finer tungsten particles comparable in size to the starting tungsten powder size (i.e., 5 to 10 microns). In solid state sintering operations, processing temperatures are maintained below the solidus temperature of the alloy and therefore some degree of pressure application is necessary to bring about the densification of the powder charge. This pressure application can take the form of any number of typical extrusion processes or use the more omnidirectional compaction processes such as ROC (rapid omnidirectional compaction) (ref 15) or the Ceracon process (ref 16). Both of these latter compaction processes have recently been used in tungsten alloy development studies. Since solid state diffusional mechanisms are only operational in these compaction processes, the powder charges need to be very homogeneous if the consolidated material is to display uniform property characteristics. In the ROC study, the homogeneity of the tungsten powder charge was established by precoating the tungsten powder with nickel as the matrix material. A fluidized bed chemical vapor deposition (CVD) system was used to apply the nickel coating to the tungsten powder prior to consolidation. Starting powders as plasma spheroidized tungsten particles are shown in figure 5. These same powders nickel coated by the CVD process are shown in figure 6, and the resultant consolidated material displaying an ideal microstructure (i.e., refined grain size and near zero contiguity) are shown in figure 7. However, subsequent mechanical property evaluation showed low tensile ductilities attributed to impurity (oxygen, carbon) embrittlement at the tungsten-matrix interface. A more recent solid state sintering study employing the Ceracon process found similar low ductility values (ref 17). A number of formidible problems remain to be resolved in adapting solid state sintering processes to making good qualitiy tungsten heavy alloys. The need to perfect a precoating process that does not introduce deliterious impurities is the primary concern.

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EXPLOSIVE COMPACTION

A novel consolidation scheme that has recently been explored for tungsten material is dynamic or explosive compaction. This consolidation operation is often used when investigating ceramic powder or RSP metal powder compaction. It has recently been used for consolidating tungsten powders (refs 18 and 19) since it offers the potential of consolidating these powders into high density matrix free compacts. Various experimental compaction techniques are under study ranging from the unique high energy high rate consolidation system at the University of Texas (refs 20 and 21) that uses a homopolar electric generator to the more typical consolidation systems that use explosives. It is in this latter technique that the prospects appear more promising of obtaining sizable quantities of compacted materials in rod-shaped configurations. In such a typical experimental setup, a detonating explosive is made to drive a collapsing tube onto a can containing the powder charge. The resultant consolidated specimen is usually a solid cylinder of fully compacted powders. In the simplest of explanations the collapsing driver tube sets up a propagating shock wave through the powder charge that induces the densification, although not necessarily particle bonding. At the lowest of impacting conditions, the propagating shock wave causes sufficient plastic deformation of the particle to only densify the powder charge. Using a higher impacting velocity and correspondingly higher energy release, a sufficiently high temperature spike can accompany the wave to cause rapid particle surface melting and subsequent resolidification. More reliable interparticle bonding occurs under these circumstances of surface melting. However, the cylindrical configurations of these experiments often conspire to intensify the effects of the shock wave at the centerline to give rise to centerline porosity. Korth et al (ref 22) have been most effective to date in resolving this dilemma by special tailoring of the propagating shock wave. Furthermore, more sophisticated modeling analysis by Williamson (ref 23) has lead to better design parameter selection. The computed temperatures from this modeling at various locations in a bundle of tungsten wires that was explosively consolidated is shown in figure 8. The melting point of tungsten is noted on the figure. The analysis shows the sharp delineation of temperatures between the surface and interior of the tungsten particle to ensure only surface melting. The resultant tungsten wires experimentally compacted are shown in figure 9. Interfacial regions indicative of resolidified material are clearly seen as well as a number of voids at triple points.

Further investigative studies are needed to refine this compaction technique and establish its adequacy for tungsten powders. Presently, the presence of voids in the compacted microstructure especially prevalent in a cylindrical geometry needs to be resolved. A further post treatment using various thermomechanical techniques may be sufficient to eliminate what little residual porosity remains after the initial dynamic compaction. Another approach to consider in order to impart greater design flexibility

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into the dynamic compaction process is to precoat the tungsten particles with a compatible metal (e.g., tantalum) and heat treat to form a thin surface alloy around each particle. It is these surface alloyed tungsten particles that are then explosively compacted. The compositional gradient in the surface alloy coating now provides a broad range of reduced melting temperatures sufficient to accommodate the expected nonuniform energy deposition around the particle perimeter as the shock wave passes.

The adaptability of this explosive compaction technique to provide new generations of tungsten material is very promising. Considering that the bulk of the tungsten particle remains unaffected in this compaction process and remains relatively cool, the prospects of initially using prealloyed or dispersion strengthened particles in the compaction process appears an attractive undertaking.

MECHANICAL ALLOYING

Mechanical alloying is an emerging powder metallurgy process for metal powder production whereby elemental metal powder blends are alloyed and/or dispersion strengthened. The process is a solid state process that uses high impaction forces created between agitated balls in a high energy attrition mill. Powder particles repeatedly trapped between these impacting balls are continually cold worked, fractured, and cold welded to eventually form a homogeneous powder that at the microscopic scale is defined as a mechanically alloyed state. Many consider mechanical alloying (MA) as an alternate processing scheme to rapid solidification processing in that comparable microstructures are often produced given the same initial ingredients. The full use of MA rests upon a thorough understanding of the appropriate characteristics and properties required of the starting powder materials, a suitable processing atmosphere, and the proper rate of energy imput so as to maintain the proper balance between the opposing mechanical forces of welding and fracturing. In the aluminum system, these factors have been sufficiently researched to the point where MA aluminum alloys are commerically available (ref 24).

In a very preliminary study of mechanically alloying tungsten powders, the tungsten powders were attrited with hafnium powders; the resulting mechanically alloyed blend was then blended with nickel and iron powders and then liquid phase sintered. The resultant microstructure in contrast to a traditional blend of W-Ni-Fe powders given the same sintering conditions is shown in figure 10. Substantial grain refinement is achieved. Careful inspection of the MA sintered microstructure shows the possible presence of a third as yet undefined third phase. Further work is continuing on the processing technique for this alloy system.

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CONCLUSION

What has been presented is a brief discussion of the efforts of various groups within government organizations, academia, and industry to adapt nontraditional materials processing schemes to high density tungsten composite systems. Some of these processes have been successful in achieving fully dense bulk forms displaying unique refined microstructures and reduced contiguity. However, formidible process problems still remain unresolved with respect to developing unique mechanical properties in these alloys. It is hoped that continued efforts along these lines will result in the development of a unique generation of high density tungsten composite alloys which will better serve the needs of the DOD services.
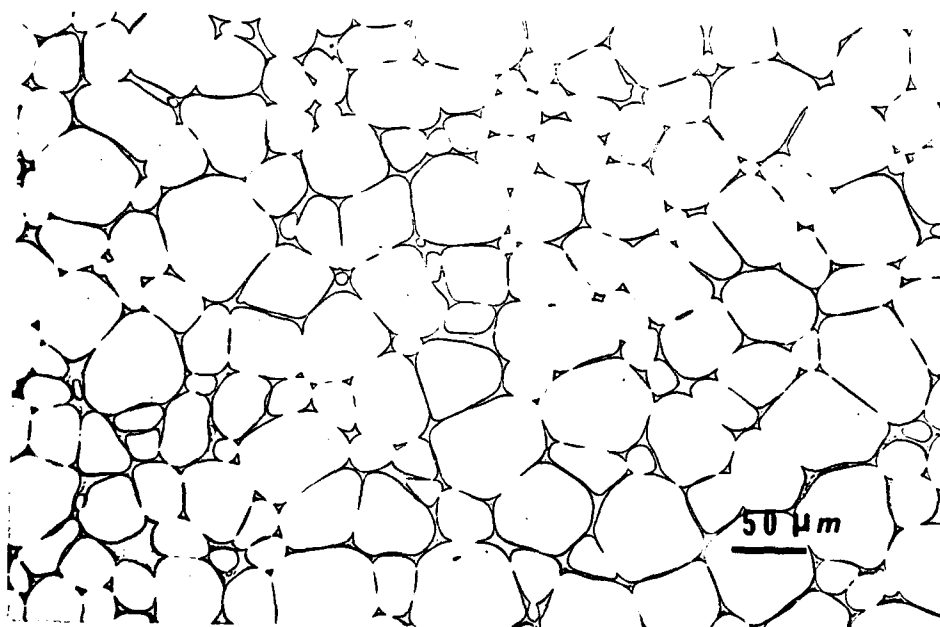
Figure 1. Typical microstructure of liquid phase sintered tungsten heavy alloy (97 wt% tungsten)

50 wt% tungsten alloy
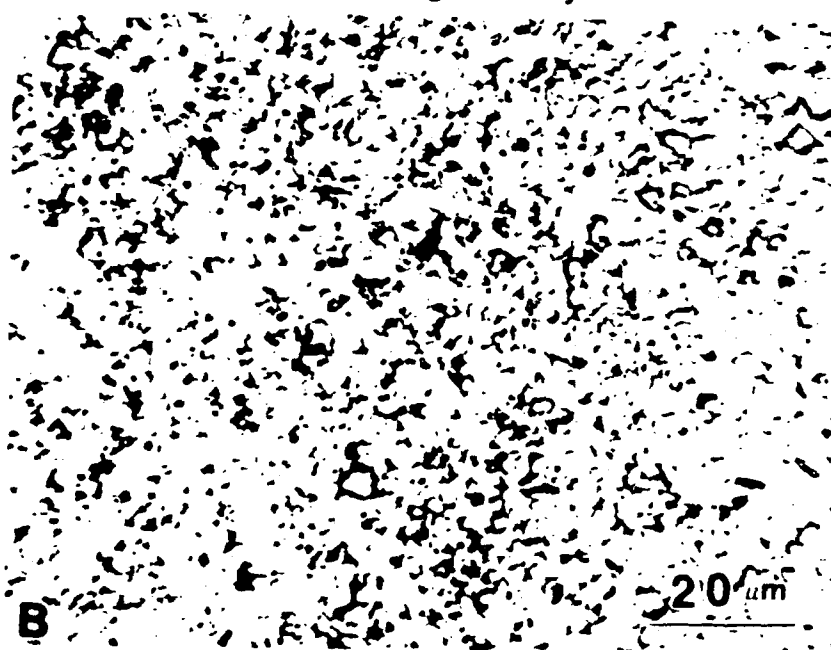
65 wt% tungsten alloy

Figure 2. Optical micrographs of as-extruded RSP tungsten alloys

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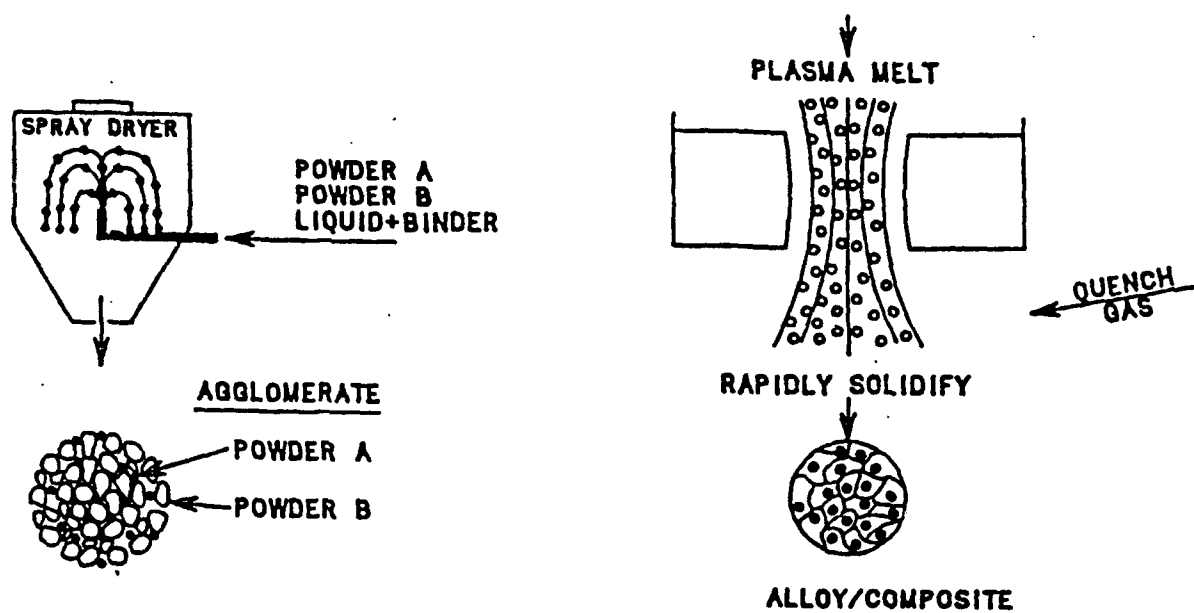
Figure 3. Plasma rapid solidification powder processing method
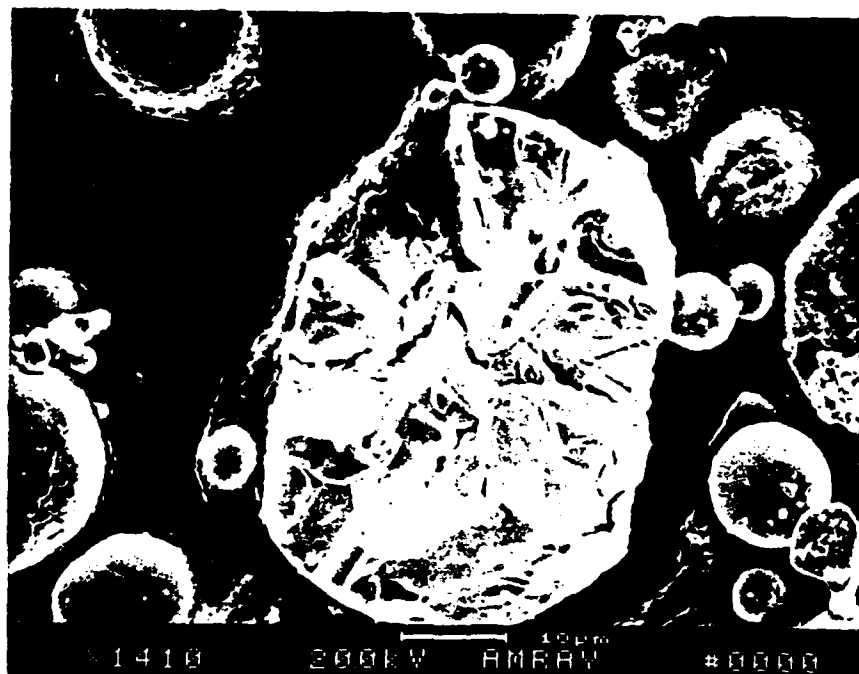

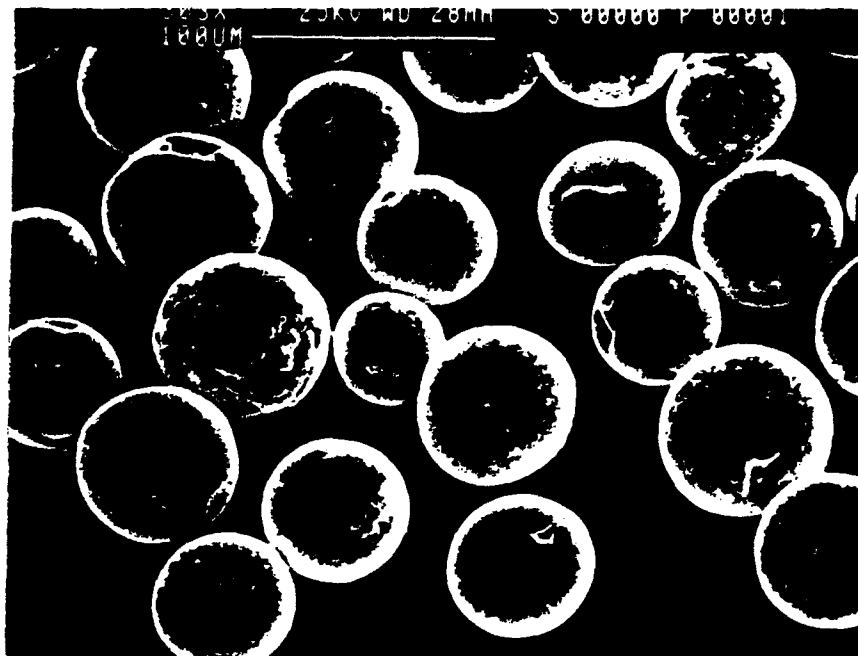Figure 4. Fractured RSP W-Hfc dispersion strengthened tungsten particle

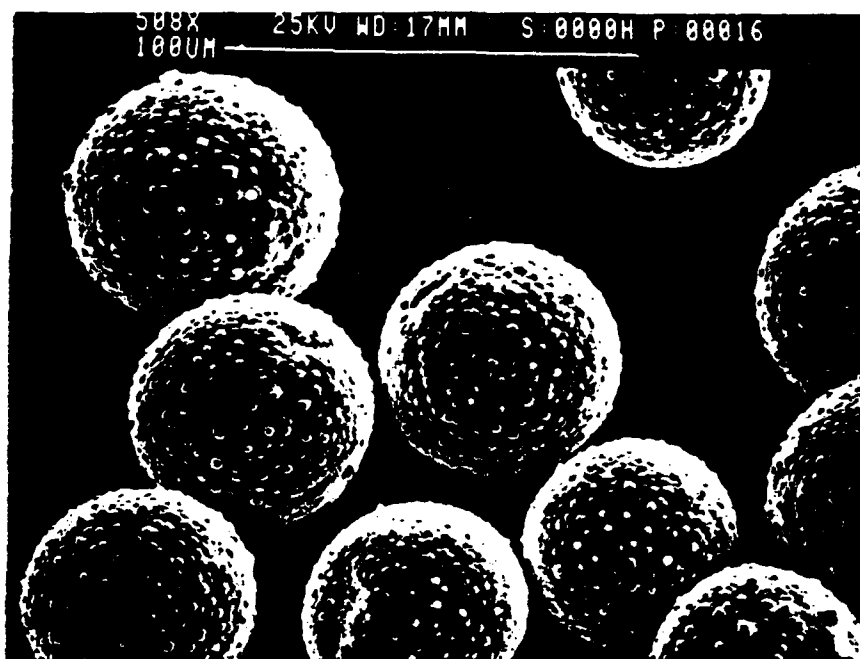Figure 5. Plasma spheroidized tungsten powder
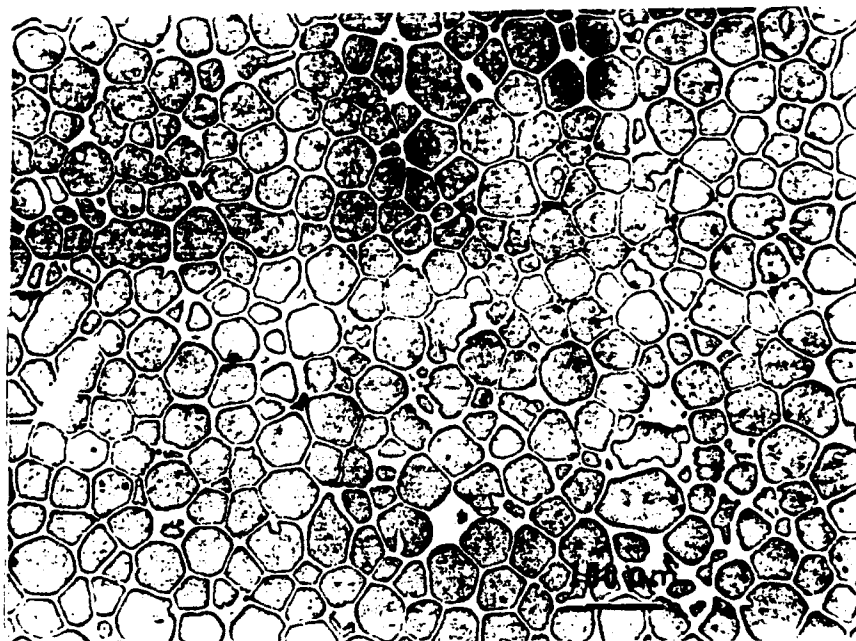

Figure 6. CVD nickel coated tungsten powder

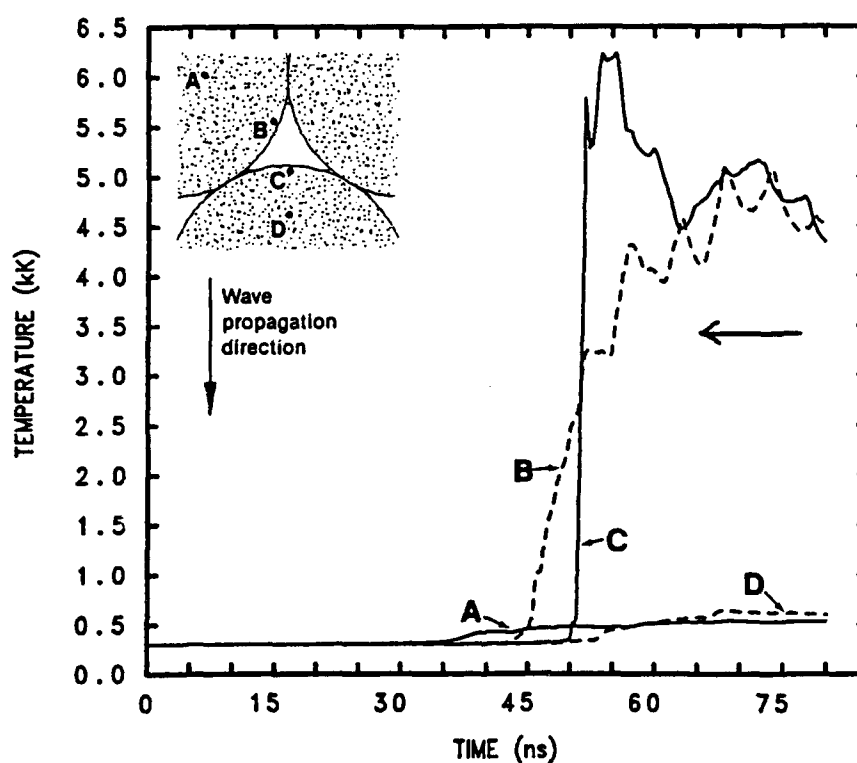Figure 7. Solid state consolidated nickel coated tungsten powders

Figure 8. Computed temperature histories after the shock at various locations in the compacted wire bundle (courtesy of G.E. Korth, INEL)
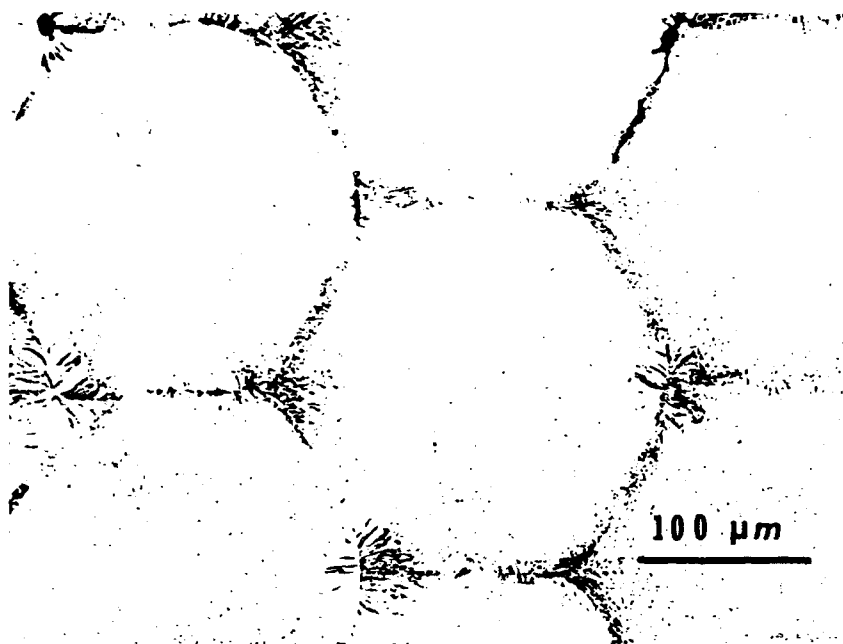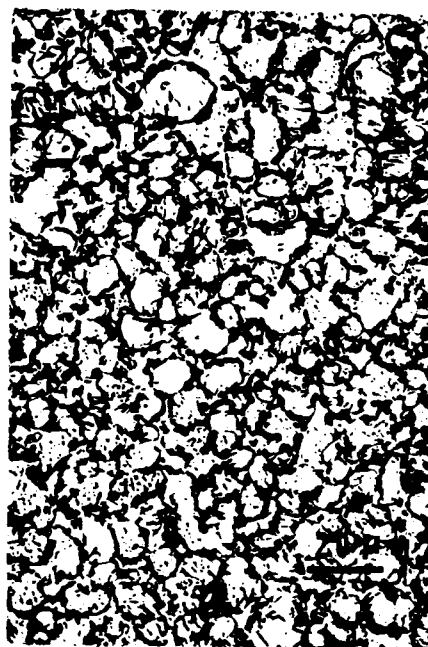
Figure 9. Transverse section of the explosively consolidated tungsten wire bundle (courtesy of G.E. Korth, INEL)

(a) 93W-4.9Ni-2.1Fe, elemental powder blend

(b) 93 (W+10Hf)-4.9Ni-2.1Fe, mechanical alloyed blended tungsten powder with hafnium

Figure 10. Liquid phase sintered 93 W alloy (sintered at 1485°C, 40 min)

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