Dynamic Consolidation and Investigation of Nanostructural W-Cu / W-Y Cylindrical Billets

To cite this article: B. Godibadze et al 2018 J. Phys.: Conf. Ser. 987 012027

View the article online for updates and enhancements.

Related content

- Tungsten copper composite fabricated by compound plastic deformation technologies
  Yang Yu and Wencong Zhang

- Fabrication of W/Cu FGM By Aqueous Tape Casting
  Shulong Liu, Qiang Shen, Guoqiang Luo et al.

- Characterization of W Coating on Cu Substrate Prepared by Double-Glow Discharge
  Zhang Fubin, Wang Zhengduo, Chen Qiang et al.
Dynamic Consolidation and Investigation of Nanostructural W-Cu / W-Y Cylindrical Billets

B.Godibadze¹, A.Dgebudze¹, E. Chagelishvili¹, G. Mamniashvili², A. Peikrishvili³

¹ G.Tsulukidze Mining Institute, 7, Mindeli St., 0186, Tbilisi, Georgia.
² Tbilisi State University, Andronikashvili Institute of Physics. 6, Tamarashvili St., 0177, Tbilisi, Georgia
³ F.Tavadze Institute of Metallurgy and Materials Science, 10 Mindeli St., 0186, Tbilisi, Georgia.

E-mail: bgodibadze@gmail.com

ABSTRACT: The main purpose of presented work is to obtain W-Cu & W-Y cylindrical bulk nanostructured billets by explosive consolidation technology (ECT) in hot condition, with low porosity near to theoretical densities and improved physical / mechanical properties. Nanocomposites were subjected to densification into cylindrical steel tube containers using hot explosive consolidation (HEC) technology to fabricate high dense cylindrical billets. The first stage: Preliminary explosive densification of the precursor powder blend is carried out at room temperature with a loading intensity up to 10GPa to increase the initial density and to activate the particle surfaces in the blend. The second stage investigation were carried out for the same already predensified billets, but consolidation were conducted in hot conditions, after heating of samples in between 940-1100°C, the intensity of loading was equal to 10GPa. Consolidated different type of W-Cu composition containing 10-40% of nanoscale W, during investigation showed that the combination of high temperatures (above 940°C) and two-stage shock wave compression was beneficial to the consolidation of the incompatible pair W-Cu composites, resulting in high densities, good integrity and good electronic properties. The structure and property of the samples obtained, depended on the sizes of tungsten particles. It was established that in comparison with W-Cu composites with coarse tungsten the application of nanoscale W precursors and depending of content of W gives different result. Tungsten is a prime material candidate for the first wall of a future fusion reactor. In this study, the microstructure and microhardness of tungsten-yttrium (W-Y) composites were investigated as a function of Y doping content (0.5÷2 wt. %). It was found that the crystallite sizes and the powder particle sizes were increased as a result of the increase of Y content. Nearly fully dense materials were obtained for W-Y alloys when the Y content was higher than 0.5 wt. %. Investigation revealed that the Y rich phases were complex (W-Y) oxides formed during the sintering process. Also very interesting to use doping chromium with yttrium-containing alloys. e.g. (W - 10÷12 Cr -0.5÷2 Y) wt. %. The extent up to which yttrium acts as an active element improving the adherence and stability of the protective Cr 2 O 3 layer formed during oxidation is assessed. The structure and characteristics of the obtained samples depends on the phase content, distribution of phases and processing parameters during explosive synthesis and consolidation. Cu – (10-30%) W powder mixtures were formed into cylindrical rods using a hot shock wave consolidation (HSWC) process. Different type of Cu - W precursor composition containing 10, 20 and 30% of nanoscale W were consolidated near theoretical density under 900°C; The loading intensity was under 10 GPa. The investigation showed that the combination of high temperatures (above 800°C) and two stage shock wave compression was beneficial to the consolidation of the W-Cu & W-Y composites, resulting in high densities, good integrity and good electronic properties.
1. INTRODUCTION

In recent years, tungsten (W)-based heavy alloys have received increased use in both commercial and industrial applications. Most heavy alloys consist of W particles embedded in matrix of other metals or their alloys such as iron, nickel, or copper [1]. In particular, W–copper (Cu) composites may have potential uses as heat dissipation materials in the microelectronics field [2], diverter plates in fusion reactors [3], or in special industrial (i.e., aerospace) applications.

Experimental studies relating the mechanical properties of samples formed from nanocrystalline precursor powders show that these ultra-fine-grained materials are fundamentally different from their normal, coarse-grained counterparts. These materials often have very unusual properties: they are ultra-hard and wear resistant, have an ideal compatibility of strength and elasticity, and are characterized by super-plasticity. When the average grain size is less than or equal to the wavelength of visible light, the material will also have unique optical, thermal, electrical and magnetic properties as well. Therefore, a decrease of the grain size and concomitant control of the defect substructure of the grains seems to be a promising way to improve properties of these materials. At present, there are various methods (e.g., cold or hot isostatic compaction in vacuum or other media) for the manufacturing of monolithic specimens using precursor powders ranging from micro- to sub-micrometer- to nanometer-sized powders (i.e., covering the visible-light spectrum from infrared to ultraviolet wavelengths).

All existing conventional technologies, alongside with imparting positive properties, introduce certain negative features. Nanometer-scale grains are very sensitive to heating; with increasing temperatures, these powders begin to grow rapidly. Typically, this grain growth is non-uniform and its overall impact causes imperfections and nonuniformity in the nanostructure and, as a result, monolithic materials formed under such conditions do not have the unique physical and mechanical properties that are otherwise would be intrinsic to nanostructured materials.

Usually, decreasing the compaction or sinter temperature during low-temperature manufacturing processing does not lead to a desirable outcome. In this case, the relatively large free surface area of the powder precludes the attainment of high-density samples. Additionally, at low temperatures, the required interfacial grain-to-grain boundaries do not form; this is especially true during the compression and consolidation of refractory and ceramic powders. Thus, the as-pressed samples are characterized with high level of porosity and, therefore, inadequate physical or mechanical properties.

Nevertheless, sufficient experience has been accumulated to provide solutions to some of the aforementioned problems. The idea is to apply high temperatures, up to 1,500 K, to the samples and carry out rapid consolidation at the hot, elevated temperature, conditions. Heating of the powders or alloys before loading assists in increasing the sample’s plasticity. As a result, common boundaries, interfacial solid solutions, intermediate layers (for the case of joining bulk alloys), and another beneficial feature form. The short heating period, \( \leq 50 \) s, prevents or suppresses grain growth processes. A further novelty of the proposed, non-conventional approach relies on the fact that the consolidation of solid samples in a cylindrical geometry from sub-micrometer and nanometer-sized W–Cu & W-Y powders is performed in two stages:

a) First stage: preliminary explosive compression of the precursor powder blend is carried out at room temperature with a loading intensity of 5-10 GPa to increase the initial density and to activate the particle surfaces in the blend;

b) Second stage: the same, already pre-densified cylindrical sample is reloaded by an primary explosive shock wave with a loading intensity of 10 GPa, but at a temperature between 800-1100°C.

It is expected that the effect of the first consolidation stage is to primarily compact the precursor powder without causing a change in its microstructure. However, unlike that in conventional compaction carried out under quasi-static conditions where the powders are subjected to elevated temperatures for extended times, the second, hot consolidation stage is performed on the pre-compact ed samples by an electric resistance heating method. The heating takes place at a high rate (about 10 to 20 K/s), reaching the process temperature in approximately 0.3 to 1 min. The shortened timescale significantly decreases the
probability of the thermally activated grain growth process (as it was demonstrated in the preliminary experiments), while the high intensity shock wave loading imparts fluidity to the grain surfaces thereby increasing their plasticity and, thus generate the particle-particle bonds that would otherwise not form under the quasi-static conditions.

In this study we undertook the consolidation of copper–20 wt.% tungsten (Cu–20W) powder mixtures into cylindrical rods using both hot shock wave consolidation (HSWC) and hot vacuum compaction (HVC) processes. Two types of Cu–W precursor compositions, one type with a nanometer-scale W and another with coarser grain sizes of > 1 μm W were consolidated to near theoretical density at 800 and 1000 °C. The shock wave loading intensity was about 10 GPa; the loading intensity during static compression was 33.9 MPa (346 kg/cm²).

The intent of the investigations was to determine if the use high temperatures and the use of two-stage shock wave processing method would be beneficial, resulting in high densities, good integrity, and good electrical properties. The effects of the distribution and precursor size of the W on the structure and property of the samples were of interest. Of further interest was a determination of the mechanical properties (elastic modulus and internal friction losses) as a function of precursor W size, processing method, and processing temperature. Lastly, the electro-magnetic properties (electrical resistivity and diamagnetic susceptibility) were measured. These results are described.

2. EXPERIMENTAL PROCEDURES

The W-Cu & W-Y precursor powder blends were prepared from commercially available elemental powders. In turn these powders were consolidated into cylindrical rods using hot shock wave consolidation (HSWC) or hot vacuum consolidation (HVC) methods. Two types of Cu–W precursor compositions with two powders having a nanometer size and two powders with grain sizes of > 1 μm were consolidated to near theoretical density. The shock wave loading intensity was about 10 GPa, whereas the loading during static HVC was 33.9 MPa (346 kg/cm²). The temperatures during compaction were 800 or 1000 °C.

The explosive compaction process is carried out using a cylindrical (axi-symmetric) scheme of dynamic loading. Typically, the shock waves are initiated with the use of industrial explosives and their mixtures with varying amounts of ammonium nitrate.

After consolidation, the HSWC samples were subjected to microstructural characterization and microhardness measurements. The electro-magnetic properties (electrical resistivity and magnetic susceptibility) along with, the mechanical properties (elastic modulus and internal friction) were also measured.

3. RESULTS AND DISCUSSION

Using scanning electron microscopy (SEM) it was found that while the nanometer powders did indeed contain nanometer-scale particles, they were heavily agglomerated into multi-micrometer agglomerates. Additionally, it was found that both powders have similar agglomerate and particle size. Figures 2(a) and 2(b) illustrate the HSWC sample microstructures of the Cu–20%W billets fabricated with nanometer-sized W shown in (a) (left column), and 5-6-μm-sized W (b) (right column), respectively, at increasing magnifications after the completion of the second-stage of HSWC at 800 °C. Based on a comparative examination of the samples, it is practically impossible to find any major difference in their microstructures. While the W appears mostly agglomerated, it seems that, in general, both HSWC samples have a uniform distribution of the two phases; that is, the W phase is uniformly distributed in the Cu matrix. Note that the larger 5-6-μm-sized W grains have undergone considerable fragmentation.
Figure 1. Compacted Cu-W composites after mechanical processing, with content different percent of tungsten.

Figure 2. Microstructures of the HSWC Cu–20%W samples at increasing magnifications; (a) - Cu–20%W (Nano) and (b) Cu–20%W (5-6 μm). Pre-consolidation temperature was 800 °C and the shock loading intensity was about 10GPa.

Figure 3. Microstructures of Sample W-1.85%Y-13.15%Cr.

Figure 4. Microstructures of Sample W-2%Y.

3.1. Electronic and Magnetic Properties
The electrical resistivity measurements for the samples consolidated by both HSWC and HVC methods were carried out by the standard four-point method and are presented in Figure 5. As it is seen from the
figure, the composites fabricated by HSWC are characterized by higher resistivity values than those obtained by the HVC method. The resistivity further decreases with increasing temperature for the HVC samples.

Based on the unusual resistivity properties of the nanometer-sized powder samples, their mechanical behavior, including the relative stiffness (elastic modulus) and loss mechanisms (internal friction) were also measured. The measurements were conducted using an acoustic spectrometer on specimens of a rectangular shape (thin plates) in which the quarter-wave bending, and vibration mode could be excited [4]. The natural frequency of vibrations was in the range of 1-5 kHz. The vibrations were excited by the electrostatic method. Measurements of the temperature dependence of the internal friction (Q') and elasticity modulus were carried out from 100 to 500 K as the samples were heated at a rate of 1 K/min. The maximum amplitude of the deformation of the vibrating specimens did not exceed ~ 10^{-6}.

Both HSWC samples show a monotonic increase of the modulus with decreasing temperature; the nanostructured sample has a much higher elastic modulus (E) than the micro-meter sized sample (at least by five times). This is in good agreement with the relaxation data for Q^{-1}. The micrometer sample also shows a loss peak at a lower temperature than the nanometer sample.

4. CONCLUSIONS
An HSWC process was used to consolidate nanostructured W precursor powders into Cu–20%W composites to near full density. The composites have better and more uniform mechanical properties and electronic characteristics than those measured for conventional (micrometer grain size) composites. Electrical resistivity measurements of the Cu–20%W composites indicate the formation of nanostructured features at the grain boundaries. This effect, manifested as a higher electrical resistivity, is more pronounced for the HSWC samples than those fabricated with the HVC method.

It was further established that the Cu–W composites containing nanometer-scale W have a stronger diamagnetic susceptibility response and are characterized with a lower dependence of the susceptibility on the applied magnetic field than composites containing micrometer grain size W or pure copper. The proposed studies are focused on synthesis, property measurements and performance evaluation of these alloys and to provide a roadmap to their commercial utilization in industry. From the commercial standpoint it must be mentioned that Nano-structural cylindrical billets of W-Y-Cr & W-Cu composites (tubes, rods. Pallets, rings) with dimensions up to ϕ= 40mm and L=250mm will be fabricated.

Fractures analysis of the tested samples (W-Y & W-Y-Cr) showed a primarily intergranular breakage between the grains boundaries. This result supports the brittle behavior observed during the tests. A
Grain morphology was observed. Coarse W grains and rounded smaller grains containing dispersed yttrium nanoparticles generally located at the grain boundaries, inhabited the grain corrosion and growth. So, in this direction we are continue study of structure and consolidation process too.

Acknowledgement:
The authors gratefully acknowledge the financial support of the Shota Rustaveli National Science Foundation (SRNSF Grant Agreement № YS15_2.2.10_101). http://www.rustaveli.org.ge/en/

References
[1] A. Bose and R. M. German. 1996. Developments in the Sintering of Tungsten Heavy Alloys. Sintering Theory and Practice. John Wiley & Sons, Inc., New York, NY. Pp.256-58
[2] R. M. German, K. F. Hens, J. L. Johnson, and Y. Bin., 1993, Powder Metallurgy Processing of Heat Dissipation Components for Microelectronic Applications. Advances in Powder Metallurgy and Particulate Materials. Specialty Materials and Composites. Proceedings of the 1993 International Conference on Powder Metallurgy and Particulate Materials, MPIF Press, Princeton, NJ. pp. 189-202
[3] J. Boscary, S. Suzuki, K. Nakamura, T. Suzuki, and M. Akiba. 1999. “Thermal Fatigue Tests on CVD– W/Cu Divertor Plates, Fusion Engineering and Design,” Proceedings of the 1997 4th International Symposium on Fusion Nuclear Technology, Vol. 39-40, Pt. A, Elsevier, Lausanne, Switzerland, pp. 537-542
[4] F. K. Akopov, N. A. Arabadzhian, N. D. Kvataya, G. S. Oniashvili, I. V. Chkhartishvili. 2004. “Magnetic and Acoustic Spectroscopies of Sintered Composite Materials,” Phys. Met. Metallogr., Vol. 97, No. 3, pp.266-268
[5] A. I. Gusev. “The Effects of Nanocrystalline State in Solids,” Usp. Fiz. Nauk, Vol. 168, 1998, pp.55-82