Mechanical and microstructural characterization of W–Cu FGM fabricated by one-step sintering method through PM route

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Abstract: Five layer W-Cu functionally graded material (FGM) for components in nuclear fusion application was fabricated by a one-step resistance sintering process, known as spark plasma sintering (SPS). In this study effect of sintering temperature (TS) on physical, mechanical and surface property was investigated. Detailed microstructural study revealed that the graded structure of the composite layers with varying composition from 0 to 100 wt% W and Cu in opposite directions could be well densified after the SPS process. It also indicates that the fine microstructure within functionally graded layers can be maintained because of short sintering time. The sample sintered at 1050°C shows more than 90% theoretical density, hardness greater than 239±5 Hv and excellent surface scratch resistance. The result demonstrates that SPS is promising and more suitable process for fabrication of W-Cu FGM.

Keywords: Mechanical properties; Functionally graded material; Plasma Facing Material; Powder metallurgy; Spark Plasma Sintering.

1. Introduction

To construct fusion reactors, developing plasma facing components (PFCs) for the divertor application is still an issue for material scientists and researchers to overcome because it will be exposed to high thermal loads (5-20 MW/m²) and play an crucial role in the realization of a fusion reactor. According to the required material characteristics for PFCs in the International Thermonuclear Experimental Reactor (ITER), Tungsten and Copper alloys are the most suitable candidate for plasma facing and heat sink materials respectively in PFCs. Tungsten has high melting temperature, high erosion sputtering threshold, low tritium retention etc. which is suitable good thermal- mechanical properties [1-2]. Moreover, low coefficient of thermal expansion makes it a promising plasma facing material. On the other hand, copper is a well-known heat sink material option due to high thermal conductivity and high coefficient of thermal expansion. [3,4] However, the large difference in melting point and coefficient of thermal expansion (CTE) of tungsten and copper makes it difficult to fabricate by conventional methods (i.e. brazing, conventional sintering, liquid infiltration etc.) and resulted thermally induced stresses at the interface. FGM can be used as interlayer and could effectively implement the mitigation of thermally induced stresses effectively which subsequently improve the component’s life.

FGM is an advanced composite material that shows continuous variation in material properties as the dimension varies. These are materials with a smooth gradient in one or more properties which is due to dependency of microstructure or chemical composition on position. The concept of property gradation can minimise thermal stresses due to difference in the CTE of W and Cu, and thus have achieved significant importance in high temperature environments applications where one component has to fulfil contrasting requirements in different positions and a joint is inappropriate. W-Cu FGM can additionally benefit due to an obvious strengthening effect, i.e. due to embedment of hard particle (W) in the soft matrix (Cu) [5]. It not only reduces the problems caused by the mismatch of the CTE and
other physical properties of both metals, but also combines the outstanding qualities of W and Cu as described above. Hence, FGM is an appropriate and reliable option as interlayer between W and Cu for (PFC) to minimize these problems when used as interlayer.

Several techniques have been employed previously like chemical vapour deposition, plasma spraying slip casting etc. for the fabrication of PFCs, but they are expensive, complex and give porous microstructure to the FGM [6-11]. In recent decades, SPS process has gained heavy attention of the researchers and widely used for fabrication of FGM, inter metallic compounds, ceramics, and composites etc., because of ease in this process as compared to common sintering methods. SPS offers significant advantages with various kinds of new materials and consistently produces a highly dense compact due to rapid consolidation technique than conventional methods. Due to higher rate of heating and less sintering time grain growth is obstructed which will consequently keep the microstructure fine and ultimately provide better irradiation resistance [12-17]. Therefore, in this presented study W-Cu FGM was fabricated through SPS process to achieve higher density and microstructural homogeneity in the compacts for plasma facing applications.

2. Materials and Methods

W-Cu FGM was prepared from the elements of high purity with average particle size 4.3 µm and 35.78 µm of tungsten and copper respectively. Five-layered FGM samples were fabricated at three different temperatures according to the design as shown in Fig.1 with varying wt% of W and Cu (100W, 75W-25Cu, 50W-50Cu, 25W-75Cu, 100Cu) in each layer, respectively. Thickness of layers in FGM sample was kept constant is about 1 mm.

![Fig.1 (a) Schematic of five layer stacking of W-Cu powder and (b) Fabricated FGM sample by SPS.](image)

Initially W and Cu as received powders with calculated weight ratios were mixed in a planetary ball mill (Retsch PM 400) for 10h. X’Pert PRO PANalytical’s Diffractometer (λ=1.54184 Å) was used for phase identification of the milled powders. Particle size of the powder were measured by particle size analyzer (MASTERSIZER) having water as dispersant and Scanning Electron Microscopy (SEM, Zeiss Supra-55) was used for particle morphology study.

In order to prepare bulk composite sample as shown in Fig.1 (b) with desired compositions powders were stacked layer wise (~1mm each layer) in a mould of 15 mm dia. made of graphite having height about 50mm. The sintering of the samples were done in a spark plasma sintering system (SPS Syntex, 725) at 950°C, 1000°C & 1050°C at vacuum < 10 Pa for 3 min with heating rate of 100 °C/min at a load of 60 MPa. Scratch testing (micro scratch tester, UMT-CETR, Bruker) has been performed to analyze the interlayer response toward load and ultimately the bond strength of FGM layers. The test was conducted under the progressive load from 5N to 50N on a single path with speed 0.016mm/s by diamond hard tip indenter with radius 1.5 µm.
3. Results & Discussions

3.1. Powder Analysis:

The SEM image of the milled powder and initial particle size distribution Fig. 2 is clearly shown in figures below. Fig. 2 (a) shows two types of particles big agglomerated equiaxed particles and small spherical particles. The distribution plot shows that the particle distribution of two separate range almost negligible overlapping area. The distribution plot of W shows that particle size ranges between 0.4µm and 10µm with mean particle size of about 3µm. Similarly Cu particles also distributed in a range of 1µm to 400µm with average 40µm.

![Fig.2](image_url)

**Fig. 2:** (a) FESEM micrograph of W-Cu powder milled for 10h in planetary ball mill and used for FGM synthesis (b) Graphs of showing initial size range of W-Cu powders particles before milling.

![XRD pattern](image_url)

**Fig. 3:** XRD pattern of different functionally graded layers of FGM sample sintered at 1000°C by spark plasma sintering.

Patterns observed through XRD for graded layers are shown in the Fig. 3 in different colours to distinguish the pattern of each layer. The pattern shows only W and Cu peaks, indicates that there is no new phase formation during milling and sintering process. The only difference in the intensity is observed, as expected due to difference in the composition of the layers but all the patterns looks identical. The patterns also shows little broadening of the peaks due to fine size of the particles.
3.2. Microstructural study

SEM image in Fig. 4 (a) clearly shows the different layers with distinct contrast. It also shows strong interfacial bonding between different layers of bulk FGM sample. The layers show uniformity throughout the sample and gradation of Cu and W is clearly visible and differentiable through elemental mapping Fig. 4 (b). With increase in W % in the layers the microstructure is changing in a continuous manner, as it is visible in the figures Cu is uniform as a matrix and W particles are well surrounded by the Cu.

Fig.4. SEM micrograph (a) and corresponding elemental area mapping (b) showing clear gradation pattern of Cu (Red) & W (White).

3.3 Physical and Mechanical properties:

Sintered samples were taken for density measurement and density was measured with the help of Archimedes principle and values were accounted by average from repeated experiments. The black bars in Fig. 5 exhibits the % change in relative density of W/Cu FGM samples at different sintering temperatures. It increases with increase in sintering temperature. The increase of density with increase of sintering temperature is due to better covering and filling the space between the W particles by the molten Cu-and also for the reason that at higher sintering temperature the pores filled-up by molten metal resulting increase of densification [18]. The maximum value of densification reaches about 90.5 % at the sintering temperature of 1050 °C.

Fig.5. Relative density variation and average Vickers hardness plots of sintered FGM samples at different temperatures (Tₜ).

Fig. 5 also shows the Vickers hardness of tungsten richest part in W/Cu FGM samples sintered for 3 minutes at different temperatures. The plot reveals as usual results that the hardness value increases
with increase of sintering temperature. This is due to shrinkage of the open pores and the grain boundary diffusion at higher sintering temperature resulting increase of density and hardness [19]. The hardness values are highest at 1050 °C compared to other sintering temperature.

3.4 Scratch testing:

Scratch test was conducted on the polished surface of the W-Cu FGM bulk sintered samples and results are shown in Fig.6(a&b). Scratch mark on the surface of the sample shows almost uniform as the load increase from one end to other end because the sample hardness also increase from one end to other end. Optical image of the scratch profile shows more detailed insight of the surface behaviour of FGM samples, the scratch width along the length from one end to other end was almost same. Stylus holder attached with acoustic emission (AE) sensor records the high frequency signal generated during scratching. When the material undergoes some damage, deformation, fracture triggering a sudden release of elastic energy in the form of acoustic radiations and these radiation signals are recorded as an indication of material failure. The material hardness depends upon the generated signal of acoustic emission. The scratch hardness of material at different temperature has been observed by generated signal of acoustic emission. Uniform signals of Acoustic emission through out the samples shows that uniform deformation during scratch test without interfacial hinderance and layers are intact to each other. Similar behavior was also found for the coefficient of friction (COF) curve shows that no significant change in friction behaviour, only sudden hump at the interfaces of each layer interfaces due to change in compositional gradient. Moreover, the AE plot is almost homogeneous throughout the test which also supports the bond strength factor of our fabricated FGM samples.

![Fig.6](image)

**Fig.6.** Scratch test data plots for FGM samples sintered at 1000 °C (a) and 1050 °C (b) respectively.

4. Conclusions

Five layered W/Cu FGM (100W, 75W-25Cu, 50W-50Cu, 25W-75Cu, 100Cu (wt %) samples were successfully fabricated through SPS method. Each layer (~1mm thickness) is clearly visible in the FSEM micrographs. The image contrast at interface due to compositional changes is clear and well bonded at the interface. The density and hardness values of each FGM samples shows different values and it increase with increase in sintering temperature. The bond strength study through scratch test revealed good intact between interlayers throughout the sample as supported by COF and AE plots. The optimization study shows that 1050 °C sintering temperature and pressing force 60 MPa is best possible parameters to fabricate W-Cu FGM using SPS technique for use as plasma facing component application.
5. References

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