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Joining of physical vapor-deposited metal nano-layered composites

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ABSTRACT

Joining of nanostructured metals by conventional processes, such as welding, often destroys the functionality of the material by disrupting its microstructure within the heat-affected zone. We present a lithography-based approach to joining of magnetron sputtered, nano-layered Cu/Nb composites without compromising the integrity of the nano-layered architecture by using a microstructure-preserving lap joint. We characterize the lap joint using scanning electron microscopy and nanoindentation, exploring potential variations in structure and mechanical properties within the parent material, overlap zone, and gap zone. Our work advances future applications of nanocomposite materials by paving the way towards practical, microstructure-preserving methods of joining them.

A major obstacle to the technological use of bulk nanostructured metals is lack of techniques for joining them [1]. Welding is an effective joining method for engineering alloys [2]. However, it causes massive disruptions in the microstructure of the welded material, giving rise to degradation of properties [3,4]. In nanostructured metals, i.e., ones with grain or domain dimensions below 100 nm [5,6], conventional welding may destroy the nano-architecture and properties of the material entirely [1]. We present a proof-of-principle demonstration of a joining method that does not compromise microstructure or mechanical properties in a model nanostructured metal. We focus on nano-layered composites of copper (Cu) and niobium (Nb), whose strength [7,8], radiation resistance [9,10], and fatigue limit [11] are far superior to those of pure Cu or Nb. These desirable properties are lost if the microstructure of the material is disrupted, motivating our work on microstructure-preserving approach to joining.

Cu/Nb multilayer nanocomposites have so far been synthesized in one of two ways: physical vapor deposition (PVD) [12] and accumulative roll bonding (ARB) [8,13]. ARB multilayers are likelier candidates for structural applications because they may be processed in bulk quantities. On the other hand, PVD multilayers may find uses as coatings or components in small-scale devices. Novel approaches to joining are needed for both types of materials. The present work assesses the feasibility of one of the simplest types of microstructure-preserving joints in PVD multilayers: the lap joint. In a lap joint, two components are joined by overlapping and bonding together sections of surface from each component. Our goal is to demonstrate a lap joint in a PVD Cu/Nb composite and assess its mechanical properties via nano-indentation. These findings are a first step towards the development of practical methods for joining of PVD metal composites. They also constitute a first example application of lithographic techniques in the deposition of Cu/Nb thin films.

Our approach is to intentionally synthesize a Cu/Nb thin film containing a gap. We then use magnetron sputtering to fill the gap with a section of material of identical microstructure to the surrounding film. We produce metal nano-layered composites by physical vapor deposition (PVD). Alternating layers of Cu and Nb are deposited on a Silicon (Si) wafer. The sputtered film is about 500 nm thick and contains 10 alternating layers of Cu and Nb of thickness 60 nm and 40 nm, respectively. Multilayers with such layer thicknesses have been found promising in previous investigations of radiation response and thermal stability [9]. We use two unbalanced, independently controlled magnetrons at a target-substrate working distance of 65 mm in an AJA International Orion 8 RF Sputter unit. The vacuum chamber is evacuated to a base pressure $<5 \times 10^{-8}$ Torr prior to deposition. The deposition rates applied are 0.1 nm/s and 0.5 nm/s for Nb and Cu, respectively. Scanning electron microscopy (SEM) images are obtained using a Zeiss Auriga Small Dual-Beam FIB-SEM. Cross-section samples of the film are prepared by setting a cut line using a diamond scribe, placing the sample with the cut line oriented along a steel rod such as a paper clip and cutting the sample by applying manual pressure on both sides of the cut line acquiring a clean vertical edge. Sputtering of nanocomposite films is nowadays routine [10,11]. However, Cu/Nb films containing a gap of specific width have not been synthesized before.

Fig. 1 shows our ten-step lithographic process to producing a gap into a parent Cu/Nb multilayer and then joining the two sections of

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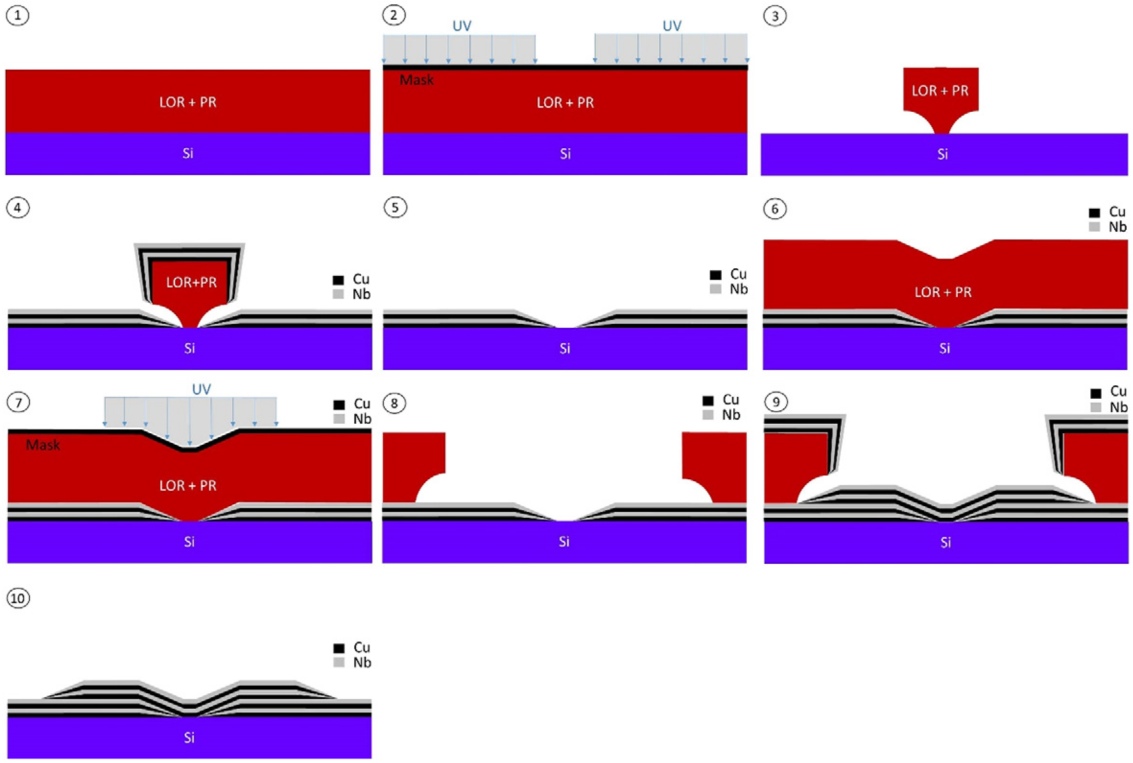

Fig. 1. 10-Step manufacturing process of a lap joint of Cu/Nb nano-layered composites.

film on either side of the gap with a lap joint. In the first step, a bi-layer lift-off-resist (LOR) and photoresist (PR), e.g. SPR3012 or AZ5214E, are deposited on a commercially available silicon (Si) wafer using a spin-coater. The LOR and photoresist bi-layer has a total thickness of approximately 3200 nm, as shown in Fig. 2a. In Step 2, the LOR and PR bi-layer is reduced to just the space of the designed gap area using photolithography and mask aligner with a 20 μm wide gap. Step 3 shows the remaining lift-off-resist (LOR) and photoresist just deposited in the intended gap area, while other parts of the photoresist are removed using MF319 or AZ726 developer. The lift-off-resist and photoresist produce a pronounced undercut, which we find to be essential for processing a clean gap. In Step 4, the Cu/Nb multilayer is sputtered onto the specimen, covering the Si wafer and the lift-off-resist and photoresist bi-layer. The schematic sketch only pictures two Cu/Nb alternating lamina, however, in our tests we sputter 10 Cu/Nb alternating lamina. The gap-infill is lifted off using acetone or remover PG at temperatures of 50 or 80 °C, respectively.

Following the foregoing steps, we obtain a Cu/Nb multilayer with a well-controlled gap, as shown in the schematic sketch of Step 5 in Fig. 1. The two ends of the nanostructured material at either side of the gap are to be bonded together without destroying the nanostructure, hence creating a metal joint with material properties as good as the parent material. The pronounced undercut produced in our approach causes the ends of the parent material to thin out while preserving the layered nanostructure. As will be shown, the absence of butt ends is beneficial to the creation of a high-quality lap joint design. In Step 6, a lift-off-resist and photoresist (SPR3012 or AZ5214E) bi-layer is applied to cover the specimen, filling the gap. The mask aligner is used to expose just the gap and overlap area with UV light, as shown in Step 7. After using acetone or remover PG, the remaining lift-off-resist and photoresist bi-layer covers everything but the gap and overlap length of the intended lap joint area, as shown in Step 8. In Step 9, Cu/Nb multilayers are once again sputtered onto the specimen. The gap is filled and an overlap is created on both sides of the parent material. Again, we find that the undercuts are fundamental for achieving thinning-out of the laminate, as shown in Step 9 of Fig. 1. There is also material sputtered onto the LOR and photoresist bi-layer. The final processing step is lift-off of the section sputtered on top of the bi-layer, as schematically shown in Step 10 in Fig. 1.

AZ5214E is an image reversal photoresist and requires flood exposure during processing. Intuitively, one might expect that gap processing might be simplified by using a positive photoresist, since it produces almost vertical ends of the parent material and a gap with a taper that opens up towards the sample surface, potentially easing the lift-off process. Indeed, that is the approach we initially attempted. However, we could not achieve an ideal lift-off using this process, likely because of friction between the parent material and gap infill material. Fig. 2b shows an example of an unsuccessful lift-off after using positive photoresist. We also found that varying pressure and temperature during lift-off does not lead to improved results.

We also explored using a negative photoresist to produce an undercut. The undercut eases the lift-off process, most likely because it provides more space for the developer to flow and distribute. However, we find that the limitation of using a negative resist is that it causes sputtered material to be deposited into the undercut, as illustrated in Fig. 2(c), which corresponds to Step 4 in Fig. 1. Residual sputtered material remains inside the gap after the lift-off process and disrupts the layered cross sectional layout, as illustrated in Fig. 2c. The final process we adopted, i.e. building the undercut with the LOR and PR bi-layer [14], circumvents all these problems and enables an easy lift-off, thinning-out of the sputtered laminate with no material residua sputtered in the undercut and opening up interesting joining possibilities.

In the final gap we synthesized, the sputtered laminate thins out continuously without a butt, as shown in Fig. 2d. The layers remain planar throughout the section and the interfaces between them appear to be atomically sharp. Hence, it is expected that the Cu/Nb layered morphology is intact and thinning out of the lamina does not impact the overall integrity within this transition zone [9]. The thinned-out ends enable a lap joint design. The lap joint design may be preferable to a butt joint design because the laminate overlap creates a higher area of contact between the parent and joint layers, potentially increasing load bearing capacity. Moreover, a gap infill for the butt joint likely