Thin Films and Coatings

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TEM microstructural characterization of co-sputtered Zr-Cu thin films metallic glasses

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The bulk metallic glasses have been studied for a long time since they present exceptional mechanical properties at room temperature and excellent corrosion resistance. A new challenge presently is to transfer such properties in the form of coatings, so called thin films metallic glasses (TFMGs). These films are, indeed, expected to be used in a large range of applications : microelectronics, optoelectronics, biomedical, and military components, besides of their properties. The overall aim of the presented study was to investigate the mechanical properties of Zr-Cu thin films (from 13 to 90 at. % Cu), deposited using a magnetron co-sputtering method. The most important issue was, in fact, to investigate the glass forming ability (GFA) of these films. A structural characterization performed by XRD at room temperature revealed that within the [33, 85] at. % Cu range films are amorphous. Nevertheless, a deeper characterization of the films was needed since some studies already mentioned the presence of crystalline domains in some TFMGs [ref]. Transmission Electron Microscopy (TEM) characterization was performed on the [33, 85] at. % Cu range films in order to confirm the amorphous structure determined by XRD. Bright field images coupled with Energy Dispersive X-Ray Spectroscopy (EDS) and Selected Area Electron Diffraction (SAED) were performed on different regions of each films. Films containing 48.3 at. % Cu were found to be fully amorphous (Fig 1). Neither on the bright field images, nor on the SAED patterns, crystalline domains were observed. For films containing less or more copper, fine crystalline nanodomains appeared (Fig 2a). Such nanocrystallised phases may be explained by the presence of Zr-Cu intermetallic compounds ($Cu_{10}Zr_7$, $Cu_{51}Zr_{14}$), pure copper or zirconia artefacts. In the case of a low content of copper, SAED patterns coupled with EDX confirm mainly the presence of zirconia. For higher copper contents, intermetallic compounds or pure copper were identified. For a Zr-59.1 at% Cu film, both copper and $Cu_{10}Zr_7$ were detected. Furthermore, these crystals are in a crystallographic relationship with a common constitutive (111) Cu plan parallel to (215) of $Cu_{10}Zr_7$. These results confirm the interest of using TEM tools to fully characterize the thin films metallic glasses. Indeed owing to X-ray detection limitations, small nanodomains cannot be detected. Thus, characterization at smaller scale is required. Mechanical properties and microstructural characterizations will now be strongly correlated in order to better understand the properties of the thin films metallic glasses. It will, thus, be possible to improve their processing and enhance their properties.

1. K. Kondoh, J. Fujita, J. Umeda, T. Serikawa, Adv. Mat. Sc. Eng., 2008 (2008) ID 518354.
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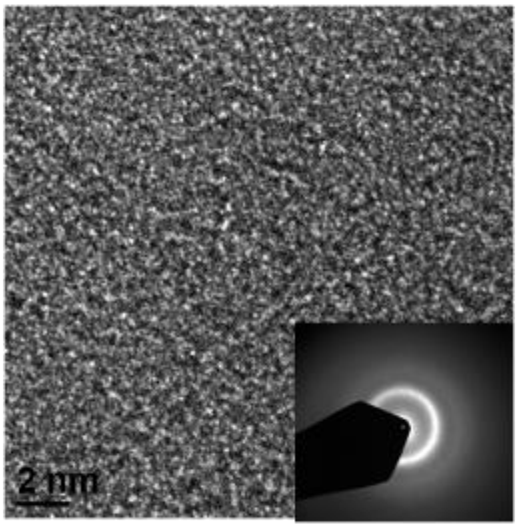

Figure 1. HRTEM plane view micrograph of Zr-48.3 at%Cu film with an inserted SAED (analyzed zone 400 nm in diameter)

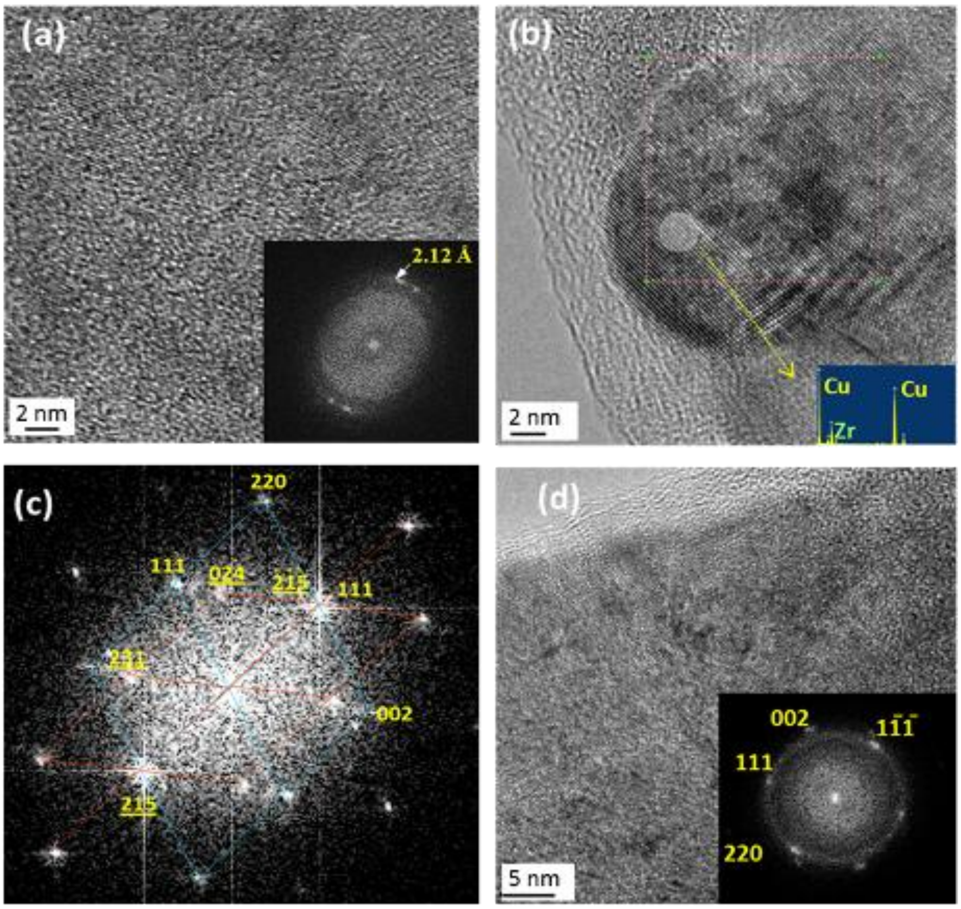

Figure 2. HRTEM characterization of different Zr-Cu thin films: a.) 40.1 at% Cu with an inserted Fourier transform diffractogram; b.) 59.1 at% Cu with an inserted EDX spectrum corresponding to the 2 nm highlighted circle ; c.) Indexed Fourier transform pattern acquired on region delimited by dotted line square of previous micrograph; non underlined numbers are corresponding to Cu indexation ([1-10] zone axis), while underlined numbers are attributed to $Cu_{10}Zr_7$ ([-742] zone axis); d.) 85.0 %Cu with an inserted Fourier transform diffractogram corresponding to copper nanocrystal