New horizons in studying intermetallics through solid state diffusion and 3D-ED:the Au-Mg system

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Phase diagrams provide indispensable guidance in the design, synthesis, processing, and application of advanced materials, including metallic alloys. In fact, they give precious information on phase stability and transformations under varying thermodynamic conditions. Among the experimental methodologies applied to study phase equilibria, the diffusion couple (DC) technique stands out as a particularly effective and versatile approach, allowing the exploration of extended compositional ranges with only few samples and avoiding kinetic effects occurring during solidification, frequently hampering the achievement of the equilibrium state. From this point of view, the DC approach also enables to discover previously undetected phases even in relatively well studied systems.

The interdiffusion region formed in DCs is traditionally characterized by electron microscopy-based techniques, such as SEM, EBSD and TEM: the first two of them are applicable to bulk samples with properly polished surfaces, instead the last one requires the extraction of thin lamellae from the layers of interest, which is usually performed by FIB/SEM. This sampling opens new horizons to solve/confirm the crystal structure of the obtained intermetallics through the recently developed, revolutionary micro-ED technique: to our knowledge this joint study was not yet exploited [1].

During our investigation of the ternary La-Au-Mg system [2], inconsistencies were observed, suggesting that a revision of Au-Mg phase equilibria was required. This binary system is well suitable for realizing the abovementioned ideas, considering that the two end-members are not oxidizable metals, easy to be shaped in form of disks and with a good interdiffusion rate at temperatures in the range 400-700 °C.

In this communication, results on the preparation and SEM/EBSD/micro-ED characterization of Au-Mg and Au-AuMg DCs at different temperatures will be presented. Several differences with the literature data were observed, including the number and composition of compounds and their occurrence as a function of temperature. The EBSD characterization revealed that at 520 °C intermediate layers are polycrystalline with small grain dimensions down to 10 μm, indicating the micro-ED as the best technique for complete structural studies. Thus, it was applied to a lamella extracted from the ~Au_3Mg band of a DC realized at 520 °C: the structural model proposed in the literature [3] was improved, proposing modulation features, as revealed from very weak satellite reflections visible in the electron diffraction pattern. Another interesting result concerns the relationship between the phases ~$Au_{60}Mg_{40}$ (new) and AuMg, which probably derive from a single high-temperature phase through a solid-state transformation: this is being investigated further.

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