An Analysis of Nanoindentation in a NiCoAlFeMo High Entropy Alloy Produced by Sintering

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High entropy alloys (HEA) consist of five or more elements in equiatomic or near equiatomic composition, they might exhibit unique microstructural features, such as solid solution phases and nanostructures. With the proper composition design, high entropy alloys can get excellent properties such as high strength and hardness, god ductility and resistance to wear. Some researchers have studied the high entropy alloys based on the elements and their content, processing conditions, microstructure and properties, but there are still some knowledge to be known. For a better information on the mechanical behavior of these materials with a microstructure formed by phases that are around or below the micrometric scale, the knowledge of the local mechanical properties can be carried out by techniques as nanoindentation and can represent a great advance in the understanding and prediction of its properties. The aim of this investigation was to evaluate the mechanical properties of individual phases by nanoindentation thecnique in a NiCoAlFeMo high entropy alloy produced by mechanical alloying and conventional sintering, to establish a relationship between microstructure, chemical composition and properties.

In a previous investigation, the microstructural features of a NiCoAlFeMo high entropy alloy produced by mechanical alloying and conventional sintering characterized by X-ray diffraction, scanning and transmission electron microscopy, besides microhardness (896 ± 90 HV) were reported [1]. Microhardness is a mechanical property that is not only sensitive to the chemical composition of the material but also to the size and distribution of the present phases [2]. Even though the formed phases of this alloy have a homogeneous distribution throughout the material, which is a typical effect of spinodal decomposition, it is possible to assume that the multiphase microstructure with different crystalline structures and the characteristic porosity produced during conventional sintering, are the two main effects responsible for the relatively high standard deviations of the microhardness test results.

For this reason, it was decided to perform nanoindentation tests, in order to know the properties of hardness and reduced modulus of each of the phases present in the alloy, to be able to determine the phase that possesses the greatest hardening, and to establish a relation between the chemical composition and the crystalline structure they possess. Nanoindentation studies were performed with a Berkovich (three-sided pyramidal) diamond tip using a nanomechanical test instrument (Ubi1, Hysitron, USA) equipped with in-situ Scanning Probe Microscopy (SPM) imaging. The results of nanoindentation test are presented in Table 1.

The highest hardness value was achieved by the tetragonal phase, while, the hardness of the BCC phase was higher than that of FCC phase, as expected. According to the chemical composition, the main constituent of the tetragonal phase is the Mo, with an atomic percentage greater than 45. The BCC Fe-type phase is a multi-principal solid solution of Ni, Al, Co and Fe; regarding Mo, this element has no

influence on the formation of this phase, it was detected that it is found in small percentages less than 0.5% at, while the Al content has been detected as a former of this phase. The FCC Ni-type phase is mainly composed by Fe, Co and Ni. Tsai et al. [3] reported that the hardening of the Al_{0.3}CrFe_{1.5}MnNi_{0.5} alloy occurs because after annealing at 700 °C, the dendritic region transforms from BCC to a tetragonal sigma structure of the CrFe-type. Although previously reported that the hardening of this type of alloys was due to the precipitation effect [4, 5]. By electron microscopy analysis and based on the literature, some authors have already asserted that the hardening is due to the formation of the tetragonal phase. However, in this study, in an experimental way and with conclusive nanoindentation tests that evaluated the local properties of the phases, it can be reported that, for the NiCoAlFeMo alloy, the tetragonal phase actually promotes the greatest hardening.

References:

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Figure 1. SEM-backscattered electron images of sintered NiCoAlFeMo alloy showing the (a) representative microstructure, and (b) the main phases evaluated by nanoindentation technique.

| Table 1. Nanomechanical properties of main phases in the NiCoAlFeM | Io alloy. |
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|--|-----------|

| Phase | Crystalline Structure – | Nanohardness | | $\mathbf{E}_{\mathbf{r}}(\mathbf{C}\mathbf{D}_{\mathbf{a}})$ |
|-------|-------------------------|--------------|-------|--|
| | | HV | GPa | Er (GPa) |
| Α | BCC (Fe-type) | 1133 | 11.11 | 244 |
| В | FCC (Ni-type) | 717 | 7.04 | 189 |
| С | Tetragonal (CoMo-type) | 1569 | 15.39 | 273 |