ICAM2009
11th International Conference on Advanced Materials
Rio de Janeiro Brazil
September 20 - 25

Environmental effects on chemical stability of $Fe_{0.85}Ni_{0.15}$ alloy studied at high pressures and high temperatures

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Abstract We investigated the samples of Fe and $Fe_{0.85}Ni_{0.15}$ alloy recovered after high-pressure/high-temperature treatment in a large volume press (LVP) and in a laser heated diamond anvil cell (DAC) in different chemical environments. Focused ion beam (FIB) and transmission electron microscopy (TEM) have been used for precise submicron scale probing of the chemical composition and the structure of the recovered materials. TEM and scanning electron microscope (SEM) structural and chemical analyses of prepared Fe-Ni samples are reported. The environmental effects, particularly, the carbon interaction with the studied Fe and Fe-Ni samples are discussed in the light of structural and chemical changes evidenced by scanning TEM and energy dispersive X-ray spectroscopy (EDS) measurements.

SEM and TEM studies of samples recovered after high-pressure/high-temperature experiments provide useful crystallochemical information about compounds that formed at extreme conditions in a specific chemical environment and can be quenched to the ambient pressure and temperature. Special methods of sample preparation are required, however, in order to reliably study the properties of tiny samples of high-pressure experiments. Samples size scale of the order of as small as 10 μm^3 makes impossible a traditional Ar ion milling technique for TEM-sample preparation. At present, FIB apparatus seem to be unique tools that, on the one hand, allow nano-scale sampling of the recovered material, and on the other hand provide integrity of the samples structural and microstructural properties during a preparation of thin sections suitable for TEM analysis [1,2]. The manufacturing of TEM sample section was done using FEI Dual Beam 600 system equipped with scanning electron microscope. The preparation process followed the *in-situ* lift-out procedure of TEM sample preparation (figure 1). We describe briefly the basic steps of the method of manufacturing of sample´s section for TEM analysis that had benefit of the recent advances in FIB equipments, like introduction of a micromanipulator and nanolithography. The results of structural and chemical analyses of precisely selected samples regions are presented and compared with complementary in-situ X-ray diffraction and Mössbauer measurements (figure 1e).

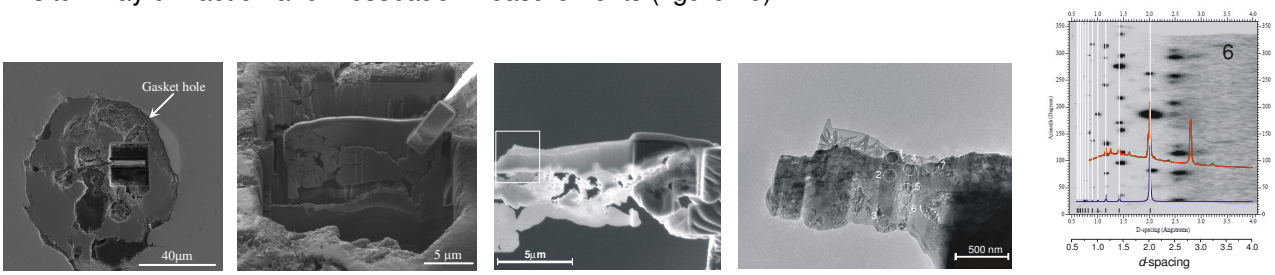

Figure 1: Images of the sample preparation sequence showing (a),(b) the extraction of the sample from the gasket, (c) the polishing of the sample fixed on the TEM grid and (d) the final TEM sample. (e) Comparison of TEM diffraction pattern collected from the selected region of the sample and integrated on azimuth angle with the in-situ X-ray diffraction measurements. The diffraction peaks from BCC phase of Fe-Ni alloy are indexed.

References

[1] M.M.H.F. Overwijk, F.C. vanden Heuvel, C.W. Bulle-Lienwma, *Nouvel scheme for the preparation of transmission electron microscopy specimens with a focused ion beam*. Journal of VacuumScience Technology, B11, 2021-2024 (1993).
[2] T. Irifune, M. Isshiki and S. Sakamoto, *Transmission electron microscope observation of the high-pressure form of magnesite retrieved from laser heated diamond anvil cell*. Earth and Planetary Science Letters, 239, 98-105 (2005).