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Crystallographic structure study of Fe₆₄Mn₃₀Si₆ shape memory alloys

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Memory Shape Alloys (MSA) are the family of materials exhibiting the Shape Memory Effect (SME), which basically allows a deformed material to obtain its previous shape after subsequent heating. It is related to a martensite-to-austenite transformation (deformation) and a reverse process during heating: $\varepsilon \leftrightarrow \gamma$ phases change. There are many systems which exhibit the SME. The most common are Ni-Ti alloys, because they are e.g. biocompatible and long term corrosion resistant [1]. The MSA play important role in industrial and medical applications. In order to reduce costs, the Fe-Mn-Si system has been studied.

In the Fe-Mn-Si material preparation process the iron (64 at.%; purity: 99,9%), manganese (30 at.%, purity: 99,9%) and silicon (6 at.%, purity: 99,9%) powders were mixed, mechanically alloyed (30 hours, 600 rpm, 1:8 ball to weight ratio, argon atmosphere), sintered (10 minutes, 900 °C temperature, 20 MPa uniaxial pressure, vacuum), and annealed (1 hour, 600 °C, vacuum). After deformation compression performed in uniaxial geometry parallel to the external stress of the sintering (room temperature, 1 mm/min, 4 % deformation). Last step was subsequent heating the samples in furnance to 200, 300, 400, 500 and 600°C, respectively.

In this study, X-Ray Diffraction (XRD) and X-ray Absorption Spectroscopy (XAS) in the XANES (X-ray Absorption Near Edge Structure) and EXAFS (Extended X-ray Absorption Fine Structure) ranges were used. XRD measurements were done on Rigaku Ultima IV (Cu-K α radiation, 0.154 nm wavelength). Synchrotron measurements were carried out at the SuperXAS beamline of the Swiss Light Source, Paul Sherrer Institute, Switzerland (partial fluorescence yield mode, room temperature).

X-Ray measurements were performed in the temperature range, starting from room temperature, up to 600°C (five temperature points) in vacuum. The results obtained are presented in Figure 1. They reveal a crystallographic structure change during heating due to the $\epsilon \rightarrow \gamma$ transformation.

Figure 2 presents results of the XAS study for the unannealed sample (Room Temperature) and after 600°C heating. Fourier transforms obtained from the EXAFS part of the Fe K-edge data, reveal one main peak at 1.96 and 2.06Å for the unannealed and 600°C annealed sample, respectively.



Figure 1. Results of XRD temperature measurements.

For the Mn K-edge EXAFS the obtained peaks are at 2.09 and 1.99Å for the unannealed and 600°C annealed sample, respectively. Small differences observed in the XANES region can be attributed to slight changes in the electronic state of the elements.



Figure 2. XANES/EXAFS results obtained for room temperature and 600°C for a) Fe and b) Mn K- α .

The results obtained show that in the case of 600°C sample the dominant phase (based on XRD) is α -BCC which is consistent with the Fe and Mn K-edge XAS (1st neighbour shell at 1.96Å and 1.99Å, respectively). In the unannealed sample the γ -phase dominates (1st neigbour shell at 2.06Å and 2.09Å, respectively).

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