A.9 : Ordering of FeCo nanocrystalline phase in FeCoNbB alloy: an anomalous diffraction study

FeCoNbB (Hitperm) alloys are important as high temperature soft magnetic materials and for high frequency applications. They have the largest induction and highest Curie temperature of both the amorphous and nanocrystalline phases among other competing nanocrystalline alloys. The soft magnetic properties of such alloy systems, where nanocrystalline grains are exchange coupled through intervening amorphous matrix are closely related to the microstructure and the composition of the amorphous matrix as well as the nanocrystalline grains. In case of FeCo alloys, the existence of ordered α'-FeCo phase (CsCl type) near equiatomic composition is of both theoretical and experimental interest e.g. the field induced uniaxial anisotropy strongly depends on the degree of atomic order, the ordered α'-FeCo alloys possess superior soft magnetic properties as compared to disordered one. Some indirect measurements such as magnetization, optical conductivity, Mössbauer spectroscopy etc. can give information about degree of atomic order of the nanocrystalline phase. However the direct tool to distinguish between the atomically ordered and disordered structure is either anomalous diffraction measurement close to an absorption edge of the constituent elements (either Fe or Co) or neutron diffraction measurement.

It is well known that the presence of superlattice (SL) reflection in the diffraction pattern is a direct confirmation of the ordered structure. SL line intensity is related to the difference between the atomic scattering factors of constituent elements. Due to similar atomic scattering factors of Fe and Co, it is very difficult to distinguished atomically ordered (simple cubic, α'-FeCo) or disordered (bcc, α'-FeCo) structure in such FeCo based alloy systems (e.g. HITPERM) using conventional diffraction technique.

Direct observation of SL reflection is possible by use of highly intense x-ray source and by choosing the wavelength close to absorption edge of the element with lower atomic number to take advantage of anomalous scattering effect. Due to dispersion corrections near an x-ray absorption edge (λ_k) there is an appreciable difference in the atomic scattering factors, f_{Fe} and f_{Co} , of Fe and Co respectively. The structure factor for the α FeCo (B2) superlattice reflection (100) is:

$$F_{hkl} = (f_{Co}^{0} - f_{Fe}^{0}) + (f_{Co}^{'} - f_{Fe}^{'}) + i(f_{Co}^{''} - f_{Fe}^{''}) = \Delta f^{0} + \Delta f^{'} + i\Delta f^{''}$$

where $f^0$is the atomic scattering factor when $\lambda << \lambda_k$ and f' is the real part and f'' is the imaginary part of the dispersion correction.

In the present work, anomalous diffraction measurements (at ADXRD beamline of Indus-2 synchrotron source) close to the K absorption edge of Fe (7.112 keV) were performed as a direct tool to distinguish between the atomically ordered and disordered structure in case of FeCo based HITPERM $Fe_{81-x}Co_xNb_7B_{12}$ alloy (x= 20.25, 27, 40.5, 54, 60.75). Prior to the measurements all the samples were isothermally annealed under vacuum atmosphere (better than 10^{-5} Torr) at temperature of 823K for 1 h. Figure A.9.1 shows the anomalous x-ray scattering data

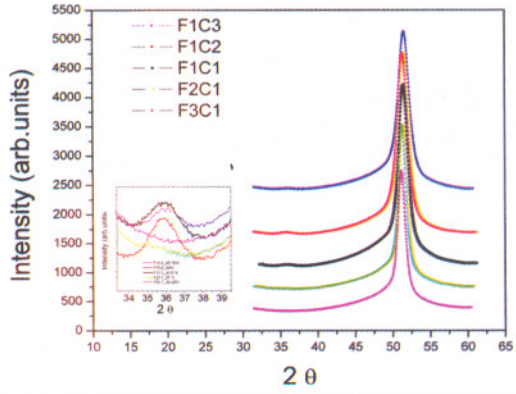

Fig.A.9.1 Anomalous diffraction measurement at Fe K -edge (7.112 keV) confirms the ordered -Fe, Co phase in samples with x≥ 40.5 % by the presence of (100) super lattice reflection

from $Fe_{81-x}Co_xNb_7B_{12}$ (x= 20.25, 27, 40.5, 54, 60.75) alloys. The presence of a (100) SL along with the fundamental line (FL), can be clearly seen in the samples with Co content ≥ 40.5 % (inset of Fig. A.9.1), which gives a direct indication of atomic ordering in such alloys. Thus this measurement clearly demonstrates that FeCo nanocrystals possess atomically ordered (B2) CsCl structure in HITPERM alloys with x=40.5 %, 54 % and 60.25 % and the absence of SL reflection in case of alloy with x=20.25 % and 27 % proves that they have an atomically disordered bcc-FeCo structure. The long range order parameter S estimated using anomalous dispersion effects as S= 0.78, 0.82 and 0.51 for the x= 40.5, 54 and 60.25 respectively. This provides unambiguous evidence that he ordering of this alloy occurs for x (Co at %) ≥ 40.5. However the alloys with x ≥ 27 are atomically disordered. Thus degree of ordering depends on the Co content in the parent alloy. Considering the superior soft magnetic properties in the ordered state of the alloys, this direct observation of the presence of atomic ordering of FeCo phase in nanocrystalline $Fe_{81-x}Co_xNb_7B_{12}$ alloys for Co concentration ≥ 40.5 is extremely important from application point of view. (For more details, please refer to *P.Gupta et al., J.Appl. Phys., 114, 083516, (2013)*

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