

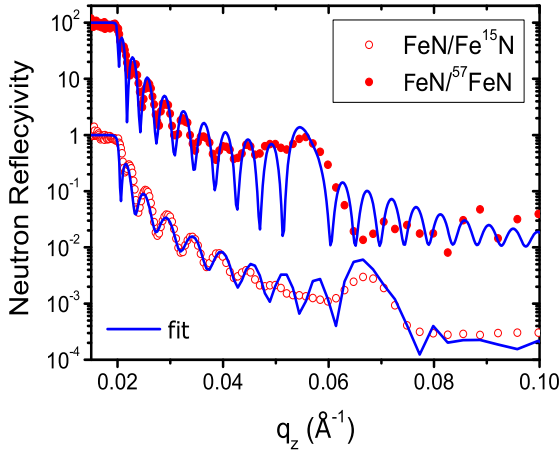
# Fe and N Self-Diffusion in Nanocrystalline FeN

M. Gupta, R. Gupta, M. Horisberger and T. Gutberlet

Laboratory for Neutron Scattering, ETH Zürich & PSI, CH-5232 Villigen PSI, Switzerland

Neutron scattering has an unique advantage due to different scattering cross sections for isotopes of an element. With the proper isotopic labelling, self-diffusion of constituents can be probed. In addition grazing incidence neutron reflectometry offers a depth resolution in sub nm range, making it possible to probe shorter diffusion lengths even at low temperatures. In the present work, nanocrystalline chemically homogenous multilayers of  $[\text{FeN}/^{57}\text{FeN}]_{10}$  and  $[\text{FeN}/\text{Fe}^{15}\text{N}]_{10}$  were prepared using magnetron sputtering and self-diffusion of both Fe and N was investigated using an *in situ* vacuum furnace at the time-of-flight neutron reflectometer AMOR.

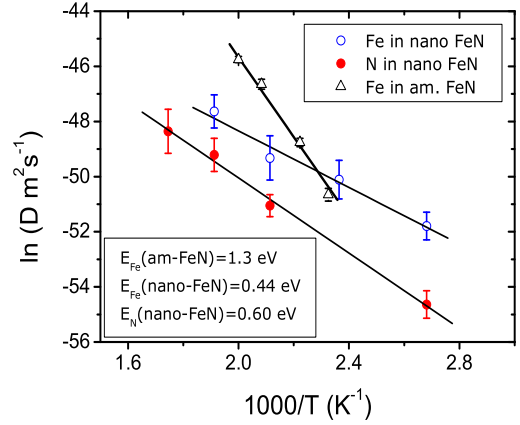
Self-diffusion in solids is a fundamental transport process. Knowledge of self-diffusion mechanism is essential for synthesis of an alloy as well as its long standing applications. A proper understanding of self-diffusion process becomes even more important when the alloys are prepared in the form of thin films due to reduced dimensionality. In a thin film or multilayer with a thickness of few nm, self-diffusion lengths in the range of sub nm have to be probed. Also, a technique having contrast for isotopes is required in order to probe self-diffusion. Neutron reflectometry is the technique best suitable for probing self-diffusion in nm range thin films and multilayers [1]. In the present report neutron reflectivity was used to probe Fe and N self-diffusion in nanocrystalline FeN prepared using magnetron sputtering[3]. Neutron scattering length for natural Fe and  $^{57}\text{Fe}$  are 9.45 and 2.3 fm, and for natural N and  $^{15}\text{N}$  9.36 and 6.6 fm, respectively. Due to distinct contrast between Fe and  $^{57}\text{Fe}$  and N and  $^{15}\text{N}$ , both Fe and N self-diffusion can be probed with proper isotopic labelling.



**Figure 1:** Neutron reflectivity pattern of  $[\text{FeN} (8 \text{ nm})/^{57}\text{FeN} (4.5 \text{ nm})]_{10}$  (upper, shifted by a factor of 100) and  $[\text{FeN} (6.4 \text{ nm})/\text{Fe}^{15}\text{N} (3.2 \text{ nm})]_{10}$  (lower) in the as-deposited state, measured at AMOR.

Multilayers of  $[\text{FeN}/^{57}\text{FeN}]_{10}$  and  $[\text{FeN}/\text{Fe}^{15}\text{N}]_{10}$  were prepared using an  $\text{Ar}+\text{N}_2$  mixture (50%  $\text{N}_2$ ) and a nanocrystalline  $\gamma$ -FeN phase was obtained. Fig. 1 shows neutron reflectivity pattern of both the multilayers and Bragg peak aris-

ing due to isotopic contrast can be seen clearly. Both the samples were annealed in the temperature range of 373-573K, for different times using a specially designed *in situ* vacuum furnace. As the multilayers are annealed both Fe and N diffuses and the Bragg peak intensity decreases. A decrease in Bragg peak intensity gives information about diffusivity and can be calculated using a procedure described in ref.[1].



**Figure 2:** Arrhenius behavior of the diffusivity in amorphous FeN (taken from [2]) and nanocrystalline FeN chemically homogeneous multilayers. The straight line fits are obtained using the relation  $D = D_0 \exp(-E/k_B T)$ .

It is interesting to observe that both Fe and N self-diffusion in nanocrystalline FeN is similar. While comparing the observed results with a previously studied amorphous FeN of similar composition, Fe diffusion in amorphous phase is significantly slower (higher activation energy, see Fig. 2). Due to presence of high density of grains and grain boundaries in nanocrystalline FeN, the diffusivity is predominantly governed by grain boundaries, resulting in an enhancement in diffusivity as compared with amorphous phase.

- [1] M. Gupta *et al*, Phys. Rev. B **70**, 184206 (2004).
- [2] M. Gupta *et al*, Phys. Rev. B **65**, 214204 (2002)
- [3] R. Gupta and M. Gupta, cond-mat/0412657, Phys. Rev. B, *Submitted*; Also in this report.

Work fully performed at SINQ  
 Proposal-number: II/04-S40  
 Instrument: AMOR