

07.5-1 ELECTRON MICROSCOPY AND X-RAY STUDY OF AMORPHOUS ALLOY $Fe_{40}Ni_{40}B_{20}$. By J.A. Zaharidova and V.C. Anchev, Department of Metallurgy, Sofia Higher Institute of Mechanical and Electrical Engineering, Sofia 1156, Bulgaria.

The crystallization of magnetic Fe-Ni-B metallic glasses has been investigated extensively in recent years. In the present paper attempts have been made to study the crystallization behaviour of amorphous alloy $Fe_{40}Ni_{40}B_{20}$. Ribbon samples of amorphous alloy $Fe_{40}Ni_{40}B_{20}$ were annealed between 573 K and 673 K under isothermal heating conditions for different times. The crystalline phases were identified using X-ray diffraction (XRD) and selected-area electron diffraction (SAD) in transmission electron microscopy (TEM).

TEM images show that a coarse dark-bright cloudy structure is typical for specimens annealed at 573 K for 3.6, 7.2, 14.4 and 28.8 ks and at 623 K for 300s. SAD and XRD results confirm the amorphous state.

Annealing at 623 K for 1.8 ks leads to changes in the amorphous matrix. The coarse cloudy dark-white contrast is more pronounced. The diffraction patterns are still those of the amorphous state, but the intensity of the first diffuse ring is increased. We can assume that a local enrichment of metallic atoms takes place during heating.

After heating at 623 K for 3.6 ks the electron microscopy shows that there are small crystallites embedded in the amorphous matrix. The last one remains in a fine grained dark-bright contrast, according to the scattered absorption. The X-ray data show that weak diffraction peaks of crystalline (Fe,Ni)-fcc solid solution and $(Fe,Ni)_3B$ orthorhombic phase appear.

After heating at 648 K for 300 s, 1.8 ks and 3.6 ks small crystallites of different quantities are observed. On the basis of XRD and SAD results, these crystallites are found to be mixture of eutectic phases - (Fe,Ni)-fcc solid solution and orthorhombic $(Fe,Ni)_3B$. Heating at 723 K for zero s leads to conservation of the amorphous state, but after heating for 1.8 ks the alloy is almost crystalline and after heating for 3.6 ks is totally crystalline in the form of a fine mixture of eutectic phases - (Fe,Ni)-fcc solid solution and orthorhombic $(Fe,Ni)_3B$.

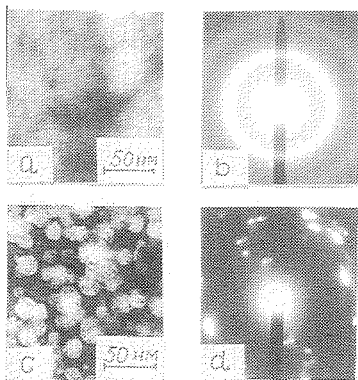


Fig.1 TEM images and SAD of samples after annealing at : a,b)623°K for 1,8 ks, c,d)648°K for 3,6ks

07.6-1 HYDROGEN ABSORPTION BY M_5X_3 PHASES. ABSTRACT. By N.J. Clark and E. Wu, School of Physical Sciences, Flinders University of South Australia, Australia.

The M_5X_3 phases crystallise in four principal structure types, namely the hexagonal Mn_5Si_3 type, the tetragonal W_5Si_3 and Cr_5B_3 types and the orthorhombic Y_5Bi_3 type.

Y_5Si_3 and Ho_5Si_3 with the $P6_3/mcm$ space group, (Mn_5Si_3 type) absorb hydrogen reversibly with production of a supercell and are sensitive to oxygen and carbon poisoning. A number of compounds with this stoichiometry which adopt the Mn_5Si_3 structure are believed to require inclusion of oxygen or carbon to stabilise the structure, (the so-called Nowotny phases), and would be expected to be inactive in hydrogen absorption. The question arises whether other M_5X_3 phases with alternative structures can absorb hydrogen.

To elucidate further the importance of structure upon hydrogen absorption, investigations of Zr_5Al_3 with the $I4/mcm$ space group have been undertaken. After activation at 100°C, Zr_5Al_3 absorbs hydrogen at room temperature and one atmosphere to form $Zr_5Al_3H_8$ accompanied by a structure change. A monoclinic cell, (space group unknown) with $a = 5.891\text{\AA}$, $b = 11.075\text{\AA}$, $c = 12.493\text{\AA}$ and $\beta = 117.68^\circ$ was determined by powder analysis. Between two and three hydrogen atoms absorb and desorb on cycling up to 150°C. The reversible absorption does not appear sensitive to oxygen contamination. When oxygen is added to reach a composition of $Zr_5Al_3O_{0.5}$ during preparation of Zr_5Al_3 a hexagonal phase with $a = 8.167\text{\AA}$ and $c = 5.680\text{\AA}$ is formed which is presumably a Nowotny phase. Unlike Y_5Si_3 this phase is not poisoned by the included oxygen and continues to exhibit significant reversible hydrogen absorption.

The implications of these observations for the positions of the oxygen atoms in the hexagonal structure and the mechanism for hydrogen absorption will be discussed.

07.7-1 STRUCTURE DETERMINATION OF POTASSIUM-BENZENE GRAPHITE INTERCALATION COMPOUND (GIC). By H. Kurata, K. Ishizuka, T. Kobayashi and N. Uyeda, Institute for Chemical Research, Kyoto University, Uji 611, Japan.

A potassium-benzene GIC (K-Bz-GIC) were prepared by the reaction of the liquid benzene with a second stage potassium GIC synthesized by the two-zone method (D.E. Nixon and G.S. Parry, J. Phys., 1968, D1, 291). The composition of the final compound can be represented as $KC_{24}(C_6H_6)_2$. Thin specimens of the K-Bz-GIC yield electron diffraction pattern showing superlattice spots from the intercalant layers superimposed on graphite spots. Two dimensional arrangement of potassium ions and benzene molecules were deduced from a Patterson map calculated only from the intercalant electron diffractions. Here, the angle of inclination of the benzene molecules had been obscure.

The electron energy loss spectroscopy (EELS) was employed to estimate the inclination angle of the benzene. The orientation dependency of an electron energy loss near edge structure (ELNES) of characteristic loss gives a wealth of information on the geometrical structure as well as electronic properties (R.D. Leapman et al., Phys. Rev., 1983, B28, 2361). The electron energy loss spectra were obtained by using JEM-100C equipped with a magnetic sector spectrometer. The orientation dependence of ELNES of carbone K edge (C_K) corresponding to the $1s \rightarrow \pi^*$ transition was analyzed. The orientation dependence of K-Bz-GIC differs from that of graphite, which clearly shows the molecular planes of benzene in K-Bz-GIC are not parallel to graphite layers. The theoretical calculations using the similar method as Leapman et al. yield a good agreement with the experimental data when the inclination angle of 35 - 40° is assumed.