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EELS ANALYSIS OF A PRECIPITATE IN V_3Si BY STEM AND HVEM

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Résumé - Nous avons effectué l'analyse d'un précipité dans V_3Si (structure A15) par spectroscopie de pertes d'énergie en microscopie électronique classique à haute tension (1000 kV) et à balayage (100 kV), par transmission. Compte tenu du diagramme de phases du système binaire V-Si, l'analyse a permis de penser qu'il s'agissait de V_5Si_3 . Ce résultat a été confirmé par microdiffraction électronique en faisceau convergent.

Abstract - The analysis of a precipitate in V_3Si (A15 structure) has been performed by transmission EELS with a HVEM (1000 kV) and with a STEM (100 kV). Taking into account the phase diagram of the V-Si system, the composition V_5Si_3 is anticipated. This result has been confirmed by convergent beam electron microdiffraction.

A new lattice defect has been found /1/ by TEM in the V_5Si_3 superconducting compound (A15 structure, Fig. 1). It looks like a couple of dislocation lines parallel to the $\langle 100 \rangle$ directions and it has been analyzed as a $a/2 \langle 100 \rangle$ faulted dipole /2/ by matching conventional electron micrographs (Fig. 2) with computer simulations. However, residual contrast has been observed /3/ in the case of perfect theoretical extinction of the faulted dipole ($\vec{g} \cdot \vec{b} = 0$, $\vec{g} \cdot \vec{b} \cdot \vec{u} = 0$ and $A = 0.97$ at room temperature). It has been first erroneously postulated /4/ that this defect could be a dispiration /5/ dipole. We have then been led to postulate the hypothesis that the faulted dipole accounted for the plastic deformation of the V_3Si matrix caused by a needle-shaped precipitate. The purpose of this communication is to report the analysis of this precipitate by Electron Energy Loss Spectroscopy (EELS) and convergent beam electron microdiffraction.

Fig. 1 - Unit cell of V_3Si (A15 structure).

I - EXPERIMENTAL PROCEDURE

The chemical analysis of the precipitate has been performed by EELS using both the 1.5 MV HVEM of the Laboratoire d'Optique Electronique (LOE) du C.N.R.S. de Toulouse operating at 1 MV /6,7/ and the 100 kV VG-HB501 STEM of the Laboratoire de Physique des Solides d'Orsay /8/. The crystallographic study of the precipitate has been undertaken with the 120 kV Philips EM400 of the L.O.E. equipped with a field emission gun.

The precipitate, which is elongated along the $\langle 100 \rangle$ directions and the rectangular cross section of which is of the order of 40-80 nm x 10-15 nm is set parallel to the incident electron beam in a $\approx (100)$ thin foil. The polishing of the samples for electron microscopy has been described elsewhere /2/.

Fig. 2 - Bright field electron micrograph (1 MV) of the lattice defects found in V_3Si by HVEM /1/.

II - EELS ANALYSIS OF THE PRECIPITATE BY HVEM

This technique has been used at 1 MV. The part of the specimen which is investigated is limited by the selecting aperture to an area of about 100 nm in diameter. Such a probe does not allow the analysis of the precipitate alone ; it includes also a part of the matrix. The presence of vanadium and silicon only has been detected, so that the precipitate belongs to the binary V-Si system, the phase diagram of which is shown in Fig. 3. In addition to this result, this technique has shown qualitatively that the precipitate is enriched in silicon with respect to the matrix. Taking into account both results, the precipitate is anticipated to be $\gamma-V_5Si_3$ /9/. This result has been confirmed by EELS with the STEM and by convergent beam electron diffraction.

Fig. 3 - The binary V-Si system phase diagram (according to /9/) showing the three compounds $\delta-V_3Si$, $\gamma-V_5Si_3$ and $\beta-VSi_2$.

Fig. 4 - Example of a 1 MV EELS spectrum on the precipitate showing clearly the vanadium L_{23} edge and the silicon K edge (enlarged 64 times with respect to the vanadium L_{23} edge) which have been used for the analysis.

III - EELS ANALYSIS OF THE PRECIPITATE BY STEM

In the STEM, the electron probe is held on a given specimen feature or scanned over a reduced area which can be adapted to the geometry and dimensions of the specimen to be analysed. In the present study, an area of 22×15 nm is investigated, of the matrix, of the correctly oriented needle-shaped precipitates or of the depressions induced by polishing around them. The EELS spectra exhibit several edges for vanadium and silicon. The two low energy edges ($V-M_{23}$ at 55 eV and $Si-L_{23}$ at 99 eV) cannot be used for quantitation because they are superimposed on an intense background which cannot be adequately represented by a power law of the form $A \cdot \Delta E^{-R}$. Consequently, one has to perform the measurements from the $V-L_{23}$ edge at 507 eV and the $Si-K$ edge at 1855 eV, as shown in Fig. 5. The atomic ratio can be estimated as follows :

$$\frac{N_V}{N_{Si}} = \frac{S_V}{S_{Si}} \times \frac{\sigma_{Si}}{\sigma_V}$$

where S denotes the signal after background stripping and σ the calculated sigma_k and signal cross section /10/ for the energy (Δ) and angular collection of detection used.

Two types of corrections have then to be taken into account, concerning the specimen itself and the instrument. The first allows for the influence of the strong thickness variations, which cause multiple scattering contributions over the edges. We use the plasmon shift correction tabulated by Stephens /11/. The second is due to the chromatic error introduced by the post-specimen objective field one electron of highly different energies ("V electrons" of ≈ 99.5 keV and "Si electrons" of ≈ 98 keV). After calibrating this effect from the known composition of the matrix, the composition of the precipitate can be established to be V_xSi with $1.4 \leq x \leq 1.6$, which provides a first clear identification that it is V_5Si_3 .

Fig. 5 - 100 kV STEM EELS spectra on a precipitate seen edge-on in V_3Si . Enlargements of the L_{23} edge of vanadium (a) and of the K edge of silicon (b) are shown, together with the extrapolation of the background.

IV - CONVERGENT BEAM ELECTRON DIFFRACTION

The crystallographic nature of the precipitate has been assessed by convergent beam electron diffraction. Fig. 6 shows a $(0\bar{1}2)^*$ diffraction pattern which confirms that the precipitate is the quadratic V_5Si_3 . The crystallographic orientation relationship between the V_5Si_3 and the V_3Si matrix is now under investigation using the convergent beam electron diffraction technique.

Fig. 6 - $(0\bar{1}2)^*$ convergent beam diffraction pattern of the precipitate seen edge-on in the V_3Si matrix. It corresponds to the quadratic V_5Si_3 phase.

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