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ALCHEMI OF $NbCr_2$/V C15-STRUCTURED LAVES PHASE

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Laves-phase intermetallics are of potential interest for use as high temperature structural materials, of which $NbCr_2$-based C15-structured Laves phases are particularly attractive.[1] Vanadium-alloyed $NbCr_2$ Laves phases have been studied.[2] The defect mechanism of a ternary Laves phase is crucial to understanding its physical metallurgy and deformation behavior.[3] It is suggested based on the Nb-Cr-V phase diagram and first-principles total energy and electronic structure calculations for $NbCr_2$ that V should occupy the B sites in C15-structured AB_2.[2] In this paper, ALCHEMI[4] is employed to examine this assumption for one composition of a V-alloyed $NbCr_2$ C15 Laves phase.

A Nb-Cr-V alloy of composition $Nb_{33}Cr_{42}V_{25}$ was prepared by arc-melting followed by annealing at 1400°C for 120 h. Specimens were prepared for microanalysis by cutting 3 mm discs followed by dimpling and ion milling. Energy-dispersive X-ray (EDX) spectra were acquired with a Philips CM30 operating at 300 kV and equipped with a Kevex Quantum detector. Fourteen spectra were collected near <014> over a range of {400} excitations between symmetry and beyond {12 0 0}. Owing to the high accelerating voltage (and therefore relatively flat Ewald sphere) used for these experiments, it was difficult to eliminate non-systematic reflections, although attempts were made to minimize this effect. Spectra were also acquired with a Philips CM12 operating at 120 kV and equipped with an EDAX superUTW detector. Nine spectra were collected near <334> over a range of {440} excitations between symmetry and {880}. Site-distributions were extracted from the data by multivariate statistical analysis (MSA)[5] with delocalization correction[6] as described elsewhere.[7,8]

The C15 Laves phase structure is essentially the same as the oxide spinel structure without oxygen. Therefore, for the compound AB_2 ($NbCr_2$ in this case) there are two distinct sublattices: A (the tetrahedral site in spinel) and B (the octahedral site in spinel). Figure 1 shows X-ray spectra collected at {400} under conditions that maximized the electron fluence on the A and B sublattices. The correlated variation of the V and Cr characteristic X-ray peaks gives qualitative evidence of similar site-distributions for these elements. However, the precision of the site-distribution extracted with MSA was poor. This imprecision can be explained by the similarity of the variations of all X-rays to the channeling, as shown in Fig. 2. Although the variation of the Nb $K\alpha$ intensity is opposite to those of the V and Cr $K\alpha$ intensities, the shapes of the three curves are similar. The three curves do not therefore vary independently, as required by MSA. The lack of site-discrimination at {400} can be attributed to the similar elastic scattering powers of the alternating planes, of composition Nb and Cr_2 in the stoichiometric binary alloy. Indeed, the intensity of the 400 reflection was relatively weak. ALCHEMI was therefore performed at the {440} systematic row, with alternating planes of composition NbCr and Cr. Here, the intensity of the 220 reflection, due entirely to scattering from the A sublattice, was pronounced. Figure 3 shows the variation of the delocalization-corrected intensity ratios[8] with orientation. The correction coefficients L_{CrX} were all within 2% of unity for the medium energy X-rays used for quantification. In contrast to the data in Fig. 2, the signals for the two host elements vary independently of one another. Multivariate statistical analysis indicated a $99.3 \pm 3.9\%$ correlation between V and Cr. The relatively large statistical error arises because the two sublattices are not completely separated onto alternating planes at {440}.[8] However, the result is consistent with exclusive site-occupancies of Nb for the A sublattice and Cr and V for the B sublattice.

In the C15 structure, the ideal ratio of atomic radii for the A and B sites is $\sqrt{3/2} \approx 1.225$. Ratios of ~1.1 to 1.6 are observed. In binary $NbCr_2$, the ratio of the Nb (2.08Å) to Cr (1.85Å) radii is ~1.13. It is expected from size effects that V (1.92Å) would substitute for Nb on the A sublattice. As the V is found to substitute for Cr, it is clear that electronic effects must be more important than size effects in this case.[9]

References

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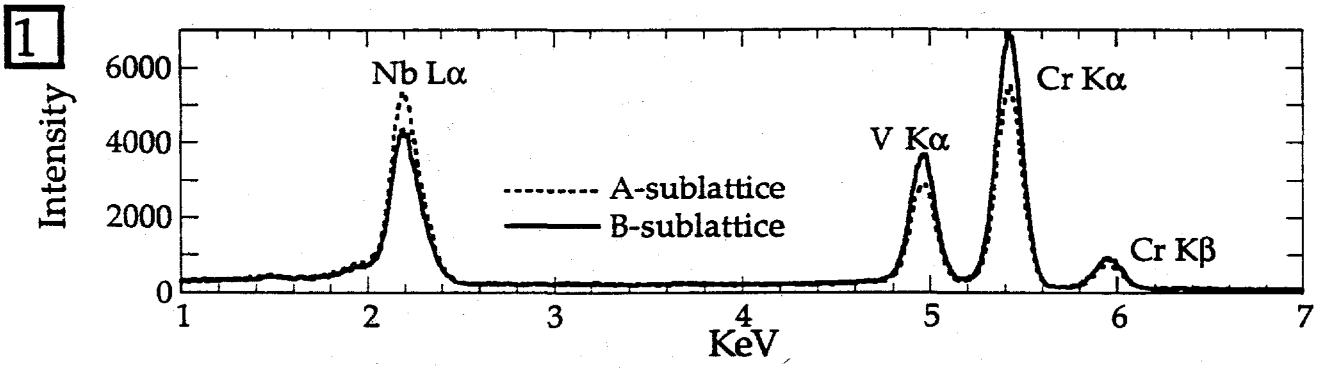

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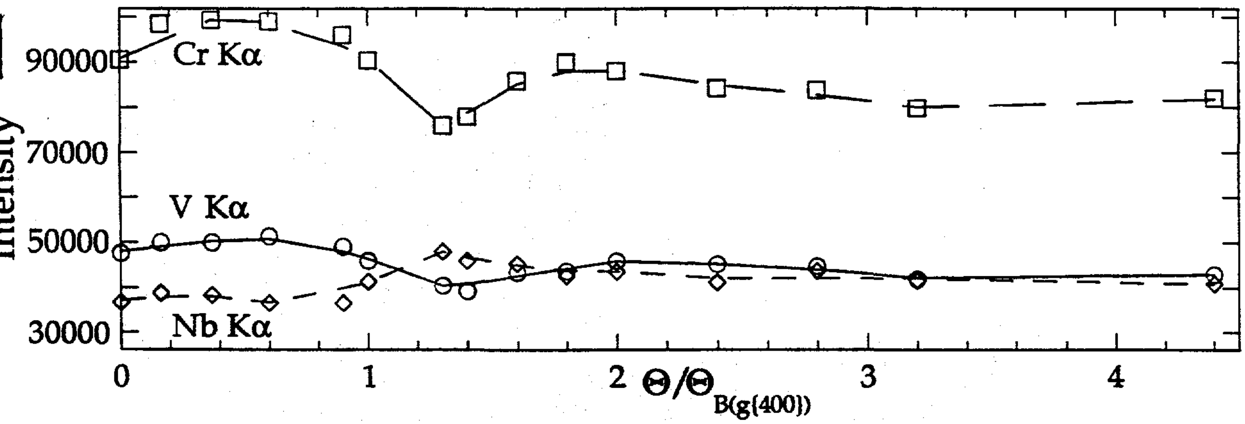

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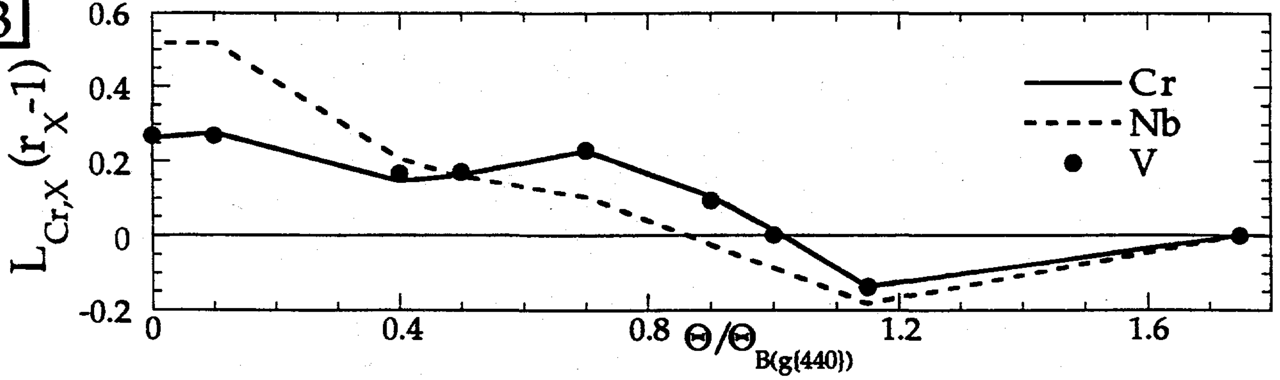

FIG 1 - EDX spectra acquired at {400} with electron fluence on the A and B sublattices.
FIG 2 - Variation of integrated X-ray intensities with orientation at the {400} systematic row.
FIG 3 - Variation of delocalization-corrected intensity ratios with orientation at the {440} systematic row.