

MICRO XAFS CHARACTERIZATION OF TRACE NICKEL DISSOLVED INTO SYNTHETIC DIAMONDS

Diamond is a unique substance possessing a high thermal conductivity, hardness and electric resistivity. The applications of diamond, valuable as hard materials, are expanding into electronics and optics. Diamond can be synthesized using either high pressure techniques or chemical vapor deposition (CVD) techniques. To produce a single large diamond crystal, however, crystallization should occur in a metallic solvent at a high temperature and high pressure. The most commonly used metallic solvents are Cr, Mn, Fe, Co, Ni, and Cu or a combination. Although many studies have examined the phase diagrams of Fe, Ni and Co with carbon at high pressures, the mechanism of diamond synthesis and the nature of the resulting synthesized diamond remain unclear.

We utilized synchrotron radiation-excited X-ray fluorescence (XRF) analysis to analyze trace impurities in synthetic diamonds. XRF analysis is

the most suitable of various analytical methods to characterize metallic impurities due to the strong elemental selectivity, high sensitivity and large capacity for spatial resolution, as compared to optical absorption or electron spin resonance (ESR). To investigate the distribution of trace impurities in diamonds grown in a $\text{Fe}_{55}\text{Ni}_{29}\text{Co}_{16}$ solvent, we utilized an X-ray microprobe system at beamline **BL39XU** [1] with a spatial resolution of 10 μm . **Figure 1** displays the Ni, Co and Fe XRF images and the transmitted X-ray image of the {110} diamond wafer of a 278 mm thickness. As previously reported [2], Ni and Co prefer to be dissolved into {111} growth sectors; the concentrations of these metals are below detection limits in {100} growth sectors. Fe cannot be detected in either the {111} or the {100} growth sectors. The Ni concentration is approximately 30 ppm. The Co concentration is an order of magnitude lower than that of Ni.

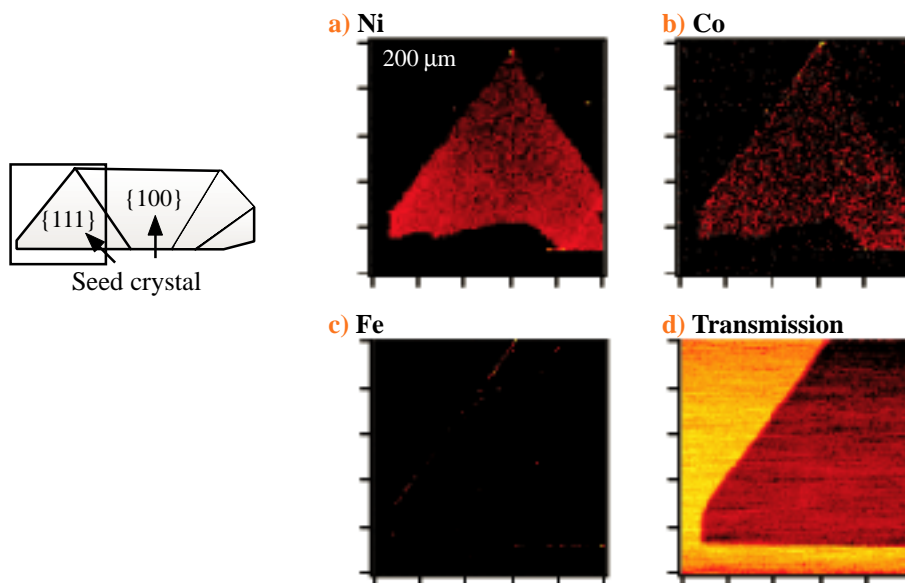


Fig. 1. Schematic drawing and X-ray images of a synthetic diamond grown at high pressure and high temperature utilizing a $\text{Fe}_{55}\text{Ni}_{29}\text{Co}_{16}$ solvent. a) Ni, b) Co, c) Fe and d) transmission images.

The combination of the brilliant undulator radiation from SPring-8 and the gap tuning method has made possible a discussion of the local structure of trace Ni and Co from X-ray absorption fine structure (XAFS) spectra. The near edge spectra of both Co and Ni display a characteristic pre-edge feature and these are completely different from the spectra of metallic Ni and Co which can be observed where inclusions exist [3]. Figure 2 shows the XAFS spectrum of Ni in the {111} growth sector. To obtain greater sensitivity, we utilized a beam size of 150 μm for these measurements. Utilizing this beam size and Ni concentration, only

4×10^{12} atoms of Ni in the diamond contributed to the XAFS spectrum. Figure 3 demonstrates the extracted EXAFS (Extended X-ray Absorption Fine Structure). Fourier transformation of the EXAFS oscillation possesses two peaks resulting from the dissolved Ni (Fig. 4). One peak, consistent with the model derived from the pre-edge peak, corresponds to Ni in the tetrahedral site of the diamond lattice. The other peak indicates the existence of Ni-Ni bond. This result suggests the presence of an extremely small metallic cluster of Ni within the diamond; further investigations are now in progress.

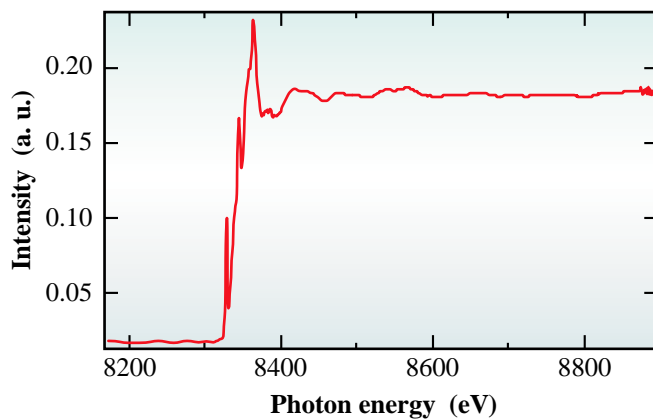


Fig. 2. Ni K XAFS spectrum obtained from dissolved Ni within the diamond using the XRF yield method. The Ni concentration is approximately 30 weight ppm (6 atomic ppm) in the {111} growth sector.

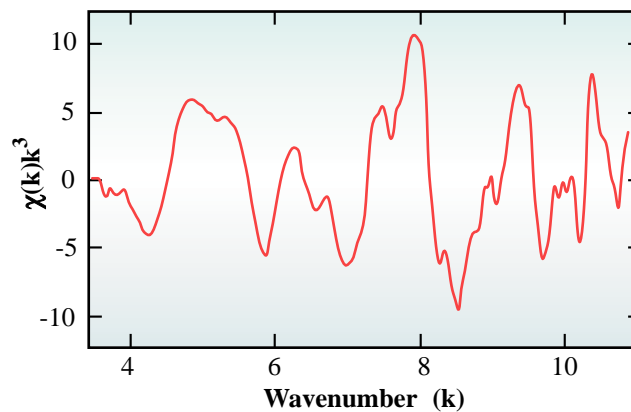


Fig. 3. EXAFS oscillations $\chi(k)$ of the dissolved Ni within the diamond.

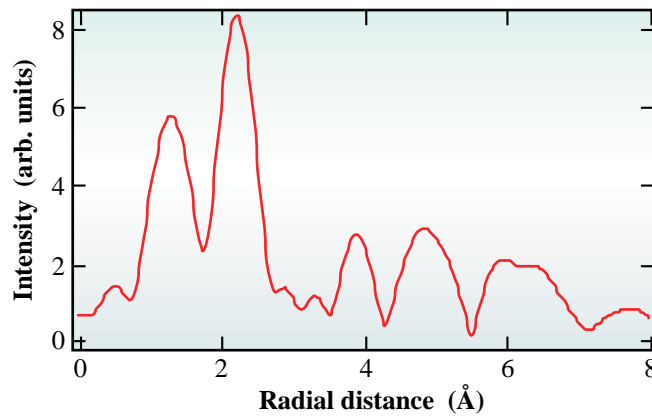


Fig. 4. Fourier transformation of EXAFS oscillations around Ni atoms within the diamond.

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