# Investigation of non-agglomerated nanodiamonds inside aluminum matrix composites produced by mechanical alloying

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Detonation-synthesized nanodiamonds (ND) are an effective type of reinforcing particle [1]. The size of the primary nanodiamond particles is 5 to 6 nm, but the particles can form agglomerates with dimensions of up to millimeter. Agglomeration is the primary barrier to the wide commercialization of nanodiamonds. For application to metal matrix composites, mechanical alloying allows effectively deagglomerating initial nanodiamond powders and producing non-agglomerated separate nanodiamond particles that are uniformly distributed in the metal matrix. For the present investigation, composites "aluminum + nanodiamonds" were used. Test specimens were prepared according to procedures described in paper [2]. The surface appearance of the produced composite granules of Al+10 vol% ND is shown in Figure 1. It is clearly observed that the agglomerates are completely shattered, and each individual nanodiamond particle is located in the matrix separately from the other particles.

Identification of non-agglomerated nanoparticles in the metal matrix becomes extremely complicated, especially if their content is less than 10 vol%; this issue considerably impedes the development of new materials. Figure 2 presents proof that the commonly used and widespread X-ray diffraction (XRD) method fails to detect non-agglomerated diamond nanoparticles 5 to 6 nm in size if they are incorporated in a metal matrix: curve 1 shows results from mixture of initial agglomerates nanodiamonds with aluminum powder (nanodiamond peaks are visible), curve 2 shows results from mechanically alloyed composite granules with non-agglomerated nanodiamonds (nanodiamond peaks are practically absent). Nuclear magnetic resonance is not available due to the electric conductivity of the material. Raman spectroscopy and X-ray photoelectron spectroscopy (XPS) can be applied only for the study of the composite granules before compaction, as their surfaces contain bare nanodiamond particles, and with a volume fraction of only 2 to 3%. It is necessary to note that in this case, the surface of the nanodiamonds can be partially covered by the matrix metal, which can cause error in the results because part of the signal from the nanoparticle can be lost, and there is no estimation of this error. After compaction of the granules, it is practically impossible to obtain a specimen surface with fully bare nanodiamond particles, as they have strong contact with the matrix and are always coated with a metal layer, although a slight one, which renders XPS and Raman spectroscopy methods unusable for the study of nanodiamonds in a metal matrix. The application of electron diffraction with a transmission electron microscope is complicated for these materials because the diamond peaks 111 and 202 are overlapped with aluminum peaks, and for a small volume fraction of nanodiamonds, the electron beam intensity is not sufficient for successful identification by diamond peak 311, due to the rather large diffraction angle.

Taking into consideration the above, it was proposed to use synchrotron radiation for the identification of non-agglomerated nanodiamonds in a metal matrix. The produced granules were consolidated by pressurizing into cylindrical specimens 5 mm in diameter and 4 mm in height. Additionally, a specimen of initial nanodiamond powder was prepared for synchrotron tests. Before

synchrotron investigation, a theoretical calculation of the expected X-ray diffraction pattern was made. Diffraction measurements were performed on the KMC-2 beamline of BESSY II (Berlin-Adlershof) in reflection geometry. A wavelength of 1.5406 Å (8048 eV) was used. A beam diameter of 0.2 mm and a Våntec 2000 area detector placed 543.6 mm from the sample were used. The detector was equipped with an anti-air scattering cone. The sample was placed at a constant angle of 45° to the beam. The diffraction angle range from 68-74° to 105°was measured. The detector was moved in 0.5° steps. As the range observed in each frame is 10°, the large degree of overlap results in the averaging-out of any potential systematic intensity fragments. The angular range was selected to observe the diamond 311 peak with a maximum separation from the surrounding peaks of the metal matrix. Depending on the volume fraction of nanodiamonds, the total measuring time was set between 1 and 6 h.

Figure 3 shows the produced synchrotron radiation X-ray diffraction patterns for the specimens of the aluminum matrix composite with 10% and 5% volume fraction of nanodiamonds. The results correspond to the theoretical predictions calculated from the crystal structures using FullProf and the Scherrer formula. The position of diamond 311 peak matches with the theoretical models and the peak position in the X-ray diffraction pattern of the pristine nanodiamonds.

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## References

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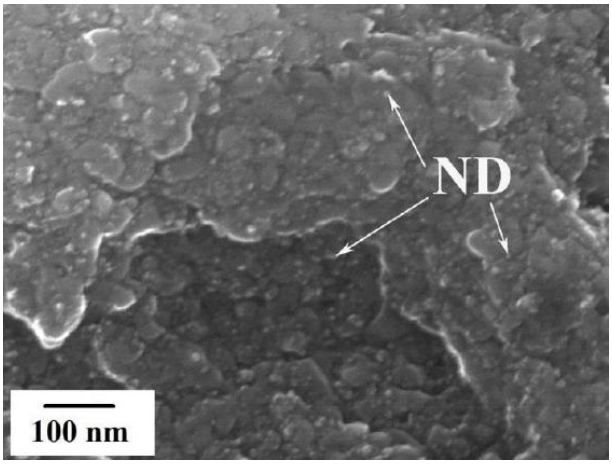


**Figure 1.** Surface appearance of Al + 10vol%ND composite granule (SEM image).

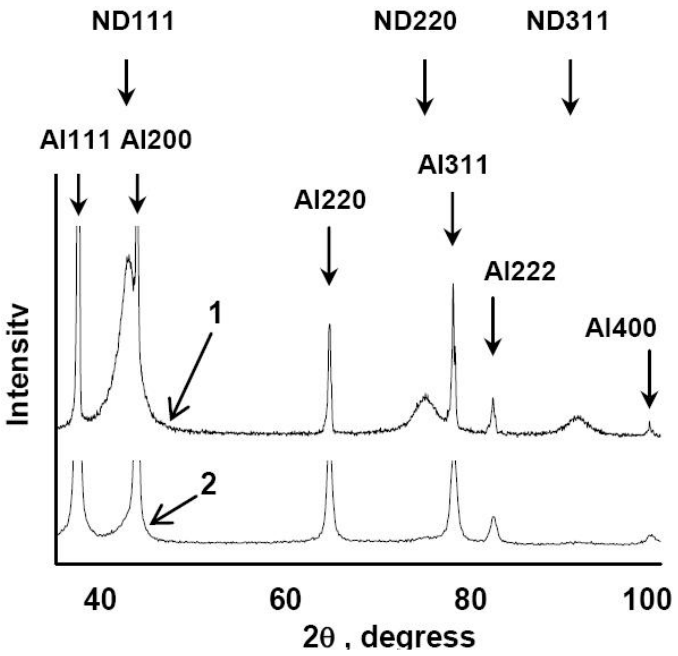


**Figure 2.** The X-ray diffraction patterns from Al+ 25%vol ND produced with use of Bruker D8 diffractometer: initial mixture of Al + ND powders (curve 1) and aluminum matrix composite after mechanical alloying during 4 hours (curve 2).

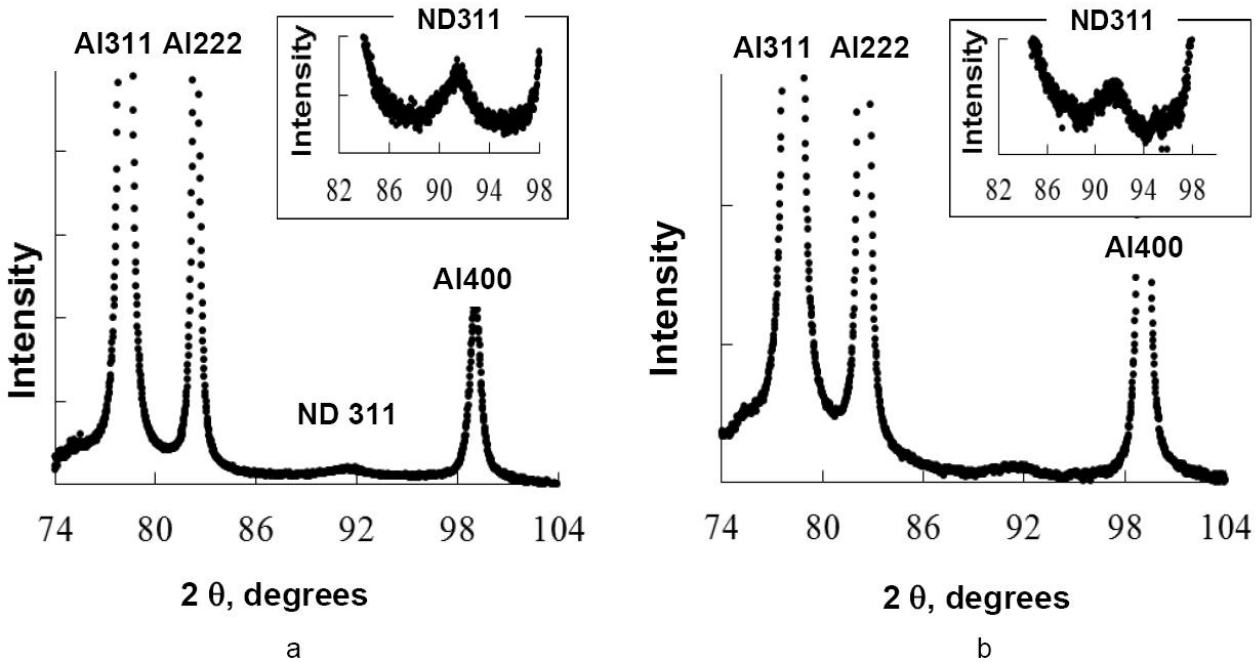


**Figure 3.** The X-ray diffraction patterns from the specimens of aluminum matrix composite with 10% (a) and 5% (b) volume fraction of nanodiamonds produced with use of synchrotron radiation.