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MICROSTRUCTURE ANALYSES OF DETONATION DIAMOND NANOPARTICLES

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14. ABSTRACT

Transmission electron microscopy and electron diffraction technique revealed detonation diamond nanoparticles approximately 5.to 6 nm in diameter. The scanning electron microscope pictures showed the octahedral shape and lustrous faces of this bulk nanomaterial. The EDAX analysis from this perfectly well purified powder material showed a single C peak. The electron diffraction and x-ray diffraction patterns confirmed these nanoparticles have octahedral crystal habit and cubic crystal system.

15. SUBJECT TERMS

Transmission electron microscope (TEM), Scanning electron microscope (SEM), and Diamond nanoparticles

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INTRODUCTION

Preparation of Detonated Nanodiamonds

Detonated nanodiamonds are produced by the *detonating* a ratio of the energetic materials, RDX and TNT, in a closed chamber. The combination of extreme temperature and pressure transform the carbon, in the energetic materials, into nanosized diamond particles. The sample is then washed out of the closed chamber using distilled water and centrifuged. This produces a mixture of diamond, carbon, and other impurities such as iron, magnesium, magnesium silicate, and magnesium oxide.

Samples, produced by NanoBlox, were submitted to the U.S. Army Armament Research, Development and Engineering Center (ARDEC), Picatinny Arsenal, New Jersey for electron microscopy analysis. The first sample was dried after being removed from the centrifuge and is designated as sample A. The next sample, sample B, was purified using acids to remove iron and magnesium compounds. Then, the graphitic carbon was eliminated via oxidation so that purified diamond nanoparticles were obtained.

Transmission Electron Microscope Specimen Preparation

The 400-mesh coated grids were used for transmission electron microscope (TEM) analyses. Powdered samples were picked up by sharp pointed tweezers and another coated 400-mesh grid was placed on top of the powdered sample, so that the sample was sandwiched between the two grids. The Philips 420 electron microscope at 120-KV voltage was used for the TEM analyses.

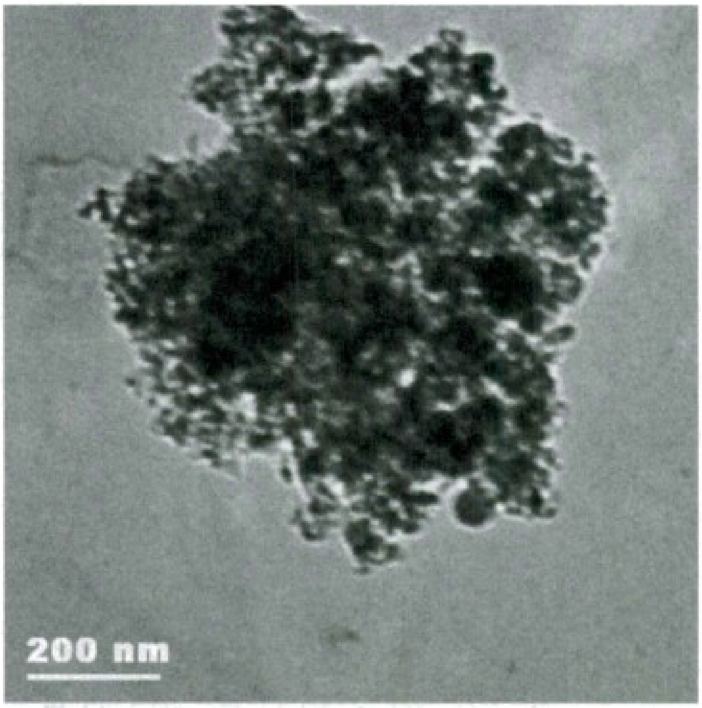
Scanning Electron Microscope Specimen Preparation

Powdered samples were taken on a circular metal holder with adhesive on top. No sputtering was used on the sample to avoid impurities transmitted on the purified sample B.

RESULTS AND DISCUSSION

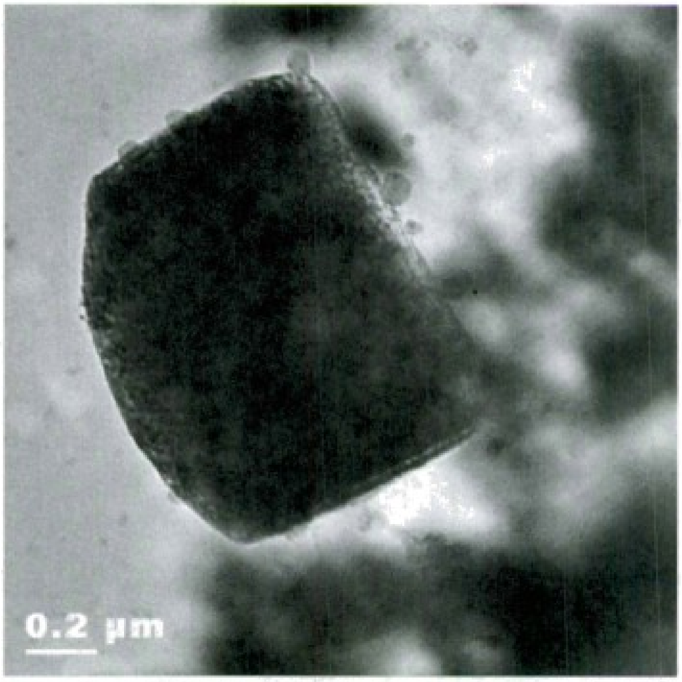
Transmission Electron Microscope Analyses

Sample A: Figures 1 to 3 are TEM pictures from the "as detonated" sample A. The selected area electron diffraction (SAED) pattern is shown in figure 4 and corresponds to the particles shown in figure 3. This diffraction pattern displays the lattice patterns from the multiple impurities found in sample A.



This sample is a mixture of magnesium aluminide, magnesium silicate, and magnesium oxide as confirmed by prior XRD analysis from Los Alamos National Labs.

Figure 1
TEM picture from as detonated sample A



This area is different, but from the same 400-mesh grid.

Figure 2
Low magnification TEM picture taken from as detonated sample A

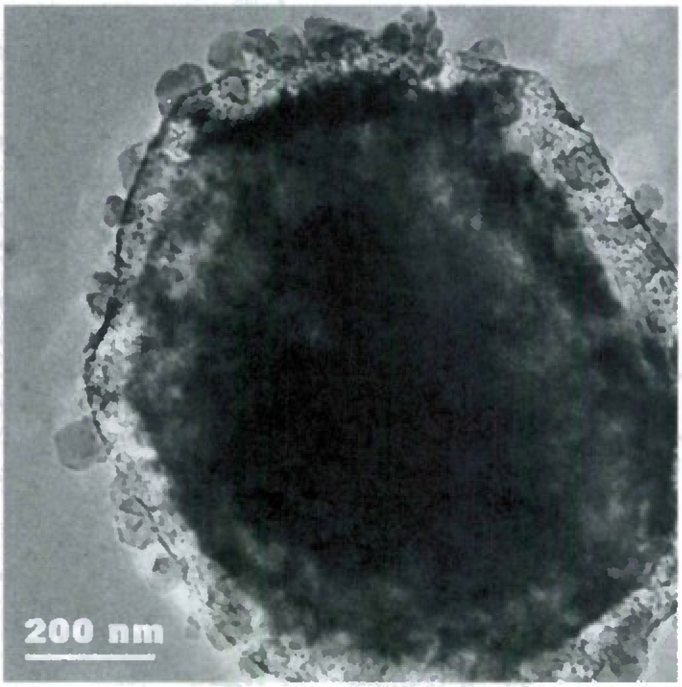
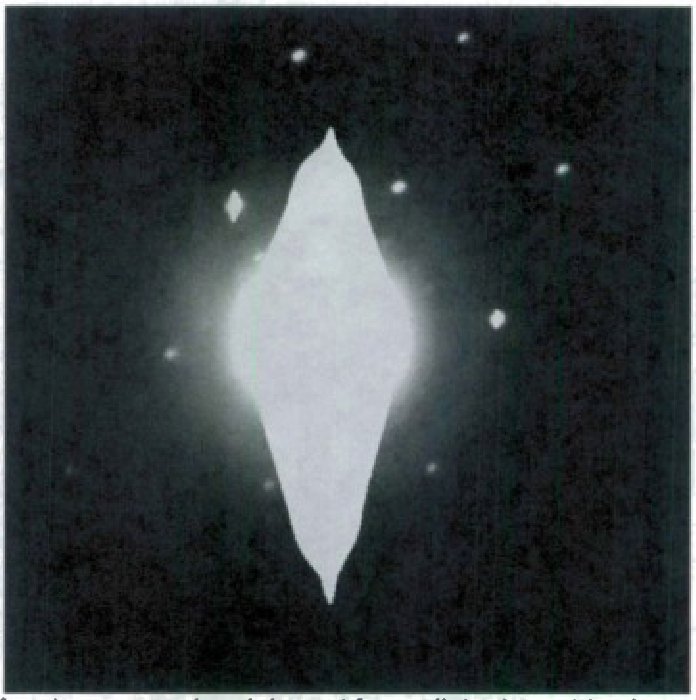


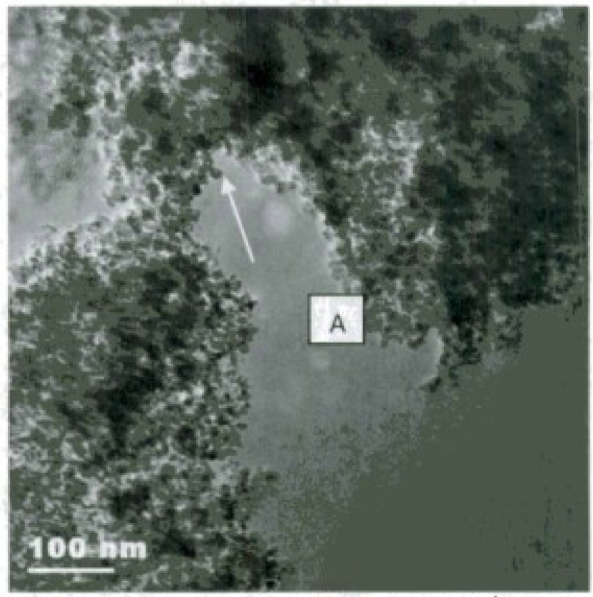
Figure 3
Decorative TEM picture obtained from as detonated specimen showing impurities plus diamond nanoparticles inside and outside of the boundary of a large crystalline material



This diffraction pattern is originated from all the impurities in sample A.

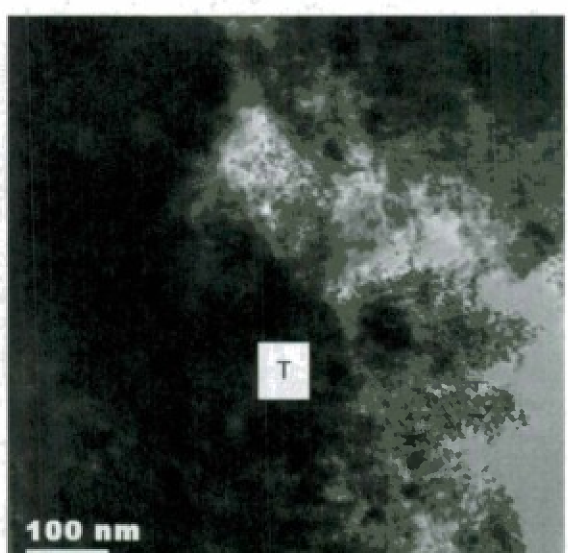
Figure 4
Selected electron diffraction pattern corresponding to the figure 3

Figure 5 shows the purified sample B and the white arrow points to a single diamond nano-particle approximately 5 to 6 nm in diameter. The area A contains multiple diamond nanoparticles. Figure 6 is another TEM micrograph of a different area of sample B. The dark dense area is due to thick powder material. Figure7 is a selected diffraction pattern from an area in figure 5. The weak reciprocal lattice point indicated by an arrow is due to the twinning effect on the (110) crystal plane.



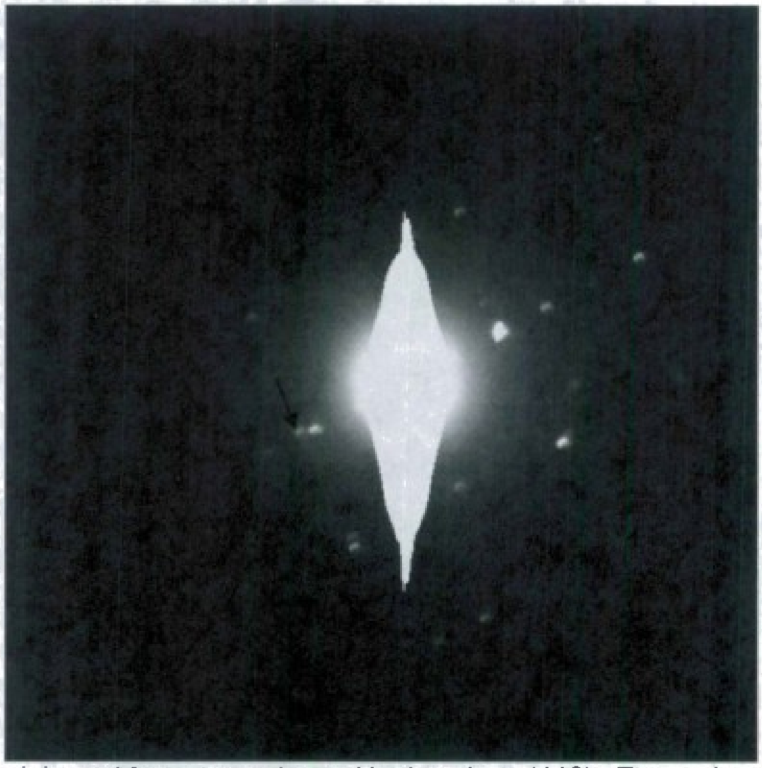
The white arrow indicates a diamond 4anoparticles. Area A resembles clusters of large numbers of nanoparticles.

Figure 5
TEM obtained from purified sample B



Dense black area T is due to thick layer of powder.

Figure 6
TEM picture taken from a different area of sample B on the same grid



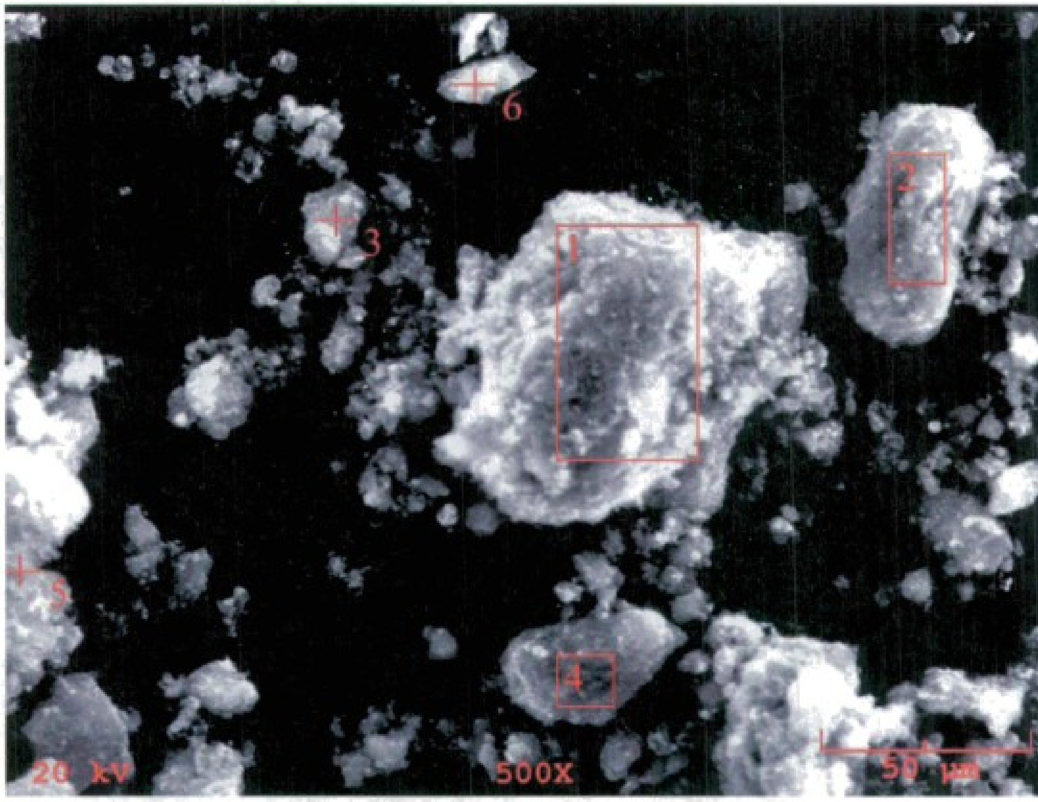
This spot pattern is originated from the reciprocal lattice plane (110). The reciprocal lattice points of low intensity as indicated by an arrow are twinning diffraction spots.

Figure 7
Selected area diffraction pattern from the area A in figure 5

Scanning Electron Microscope Analyses

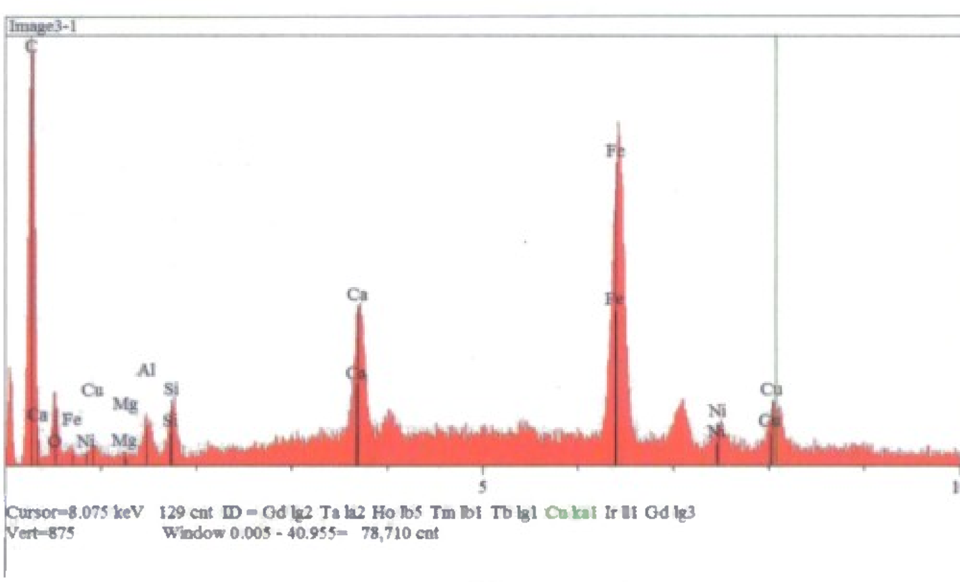
Figures 8 and 9 are scanning electron micrographs (SEMs) at magnification X500 from sample A, the as-detonated sample. The EDAX analyses are given in figures 8© and (d) and 9(b) and corresponds to the different locations of sample A that were examined. As expected, many impurities were found: iron, calcium, aluminum, magnesium, copper and silicon.

Figures 10 and 11 show particles of similar morphology, which indicates that they may have similar compositions. The EDAX analysis in figure 12 corresponds to figures 10 and 11 and shows the carbon peak only, which confirms the purification technique is perfect.



(a)

Note the selected areas 1,2,3,4 for EDAX analyses

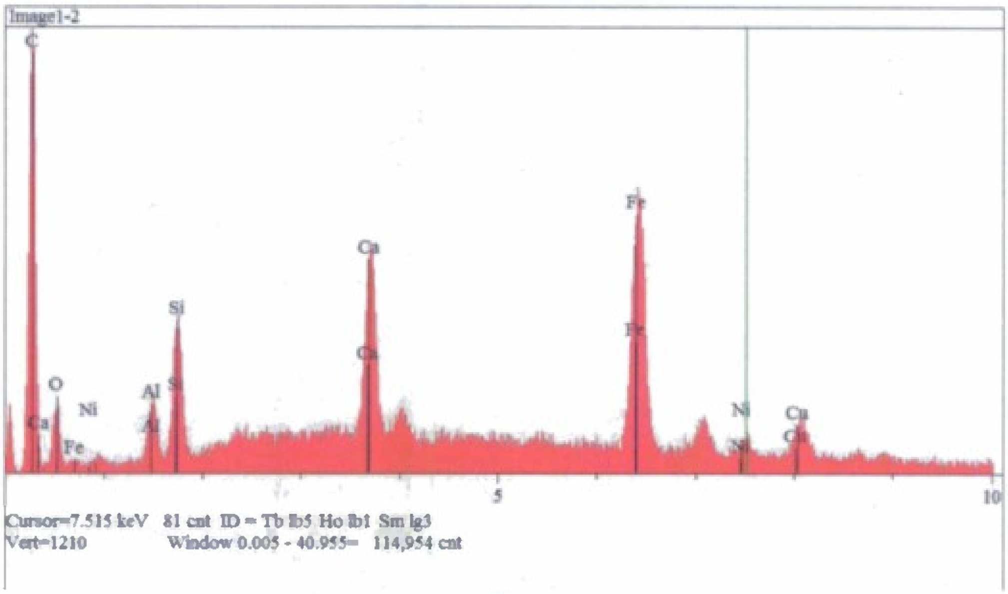


(b)

EDAX Analysis from the area 1 in (a)

Figure 8

SEM picture from as detonation sample A at low magnification (X500) showing impurities of crystals of different sizes and shapes



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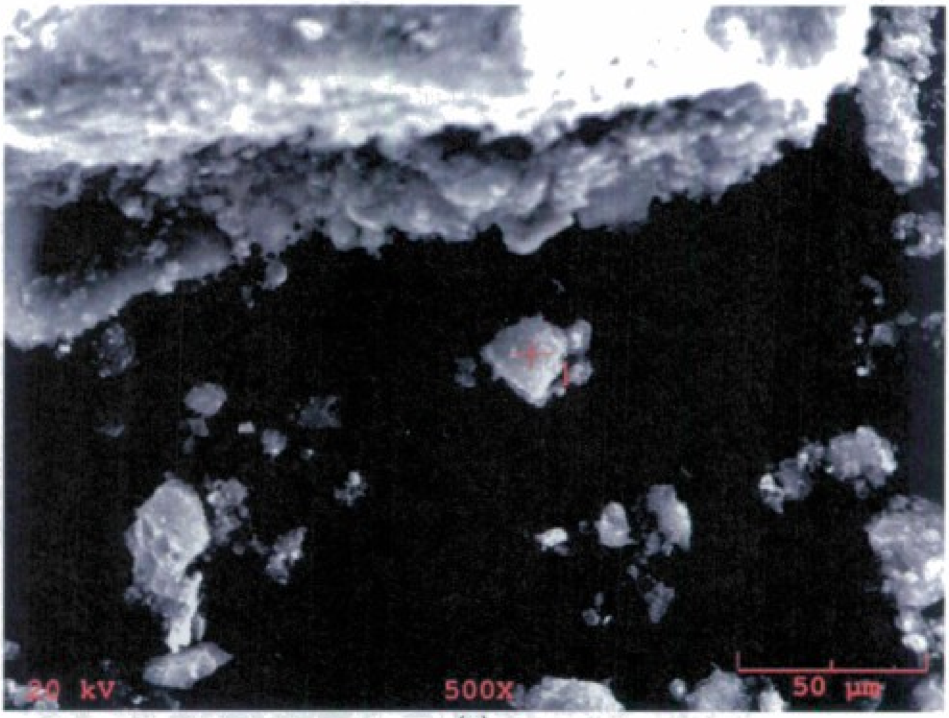
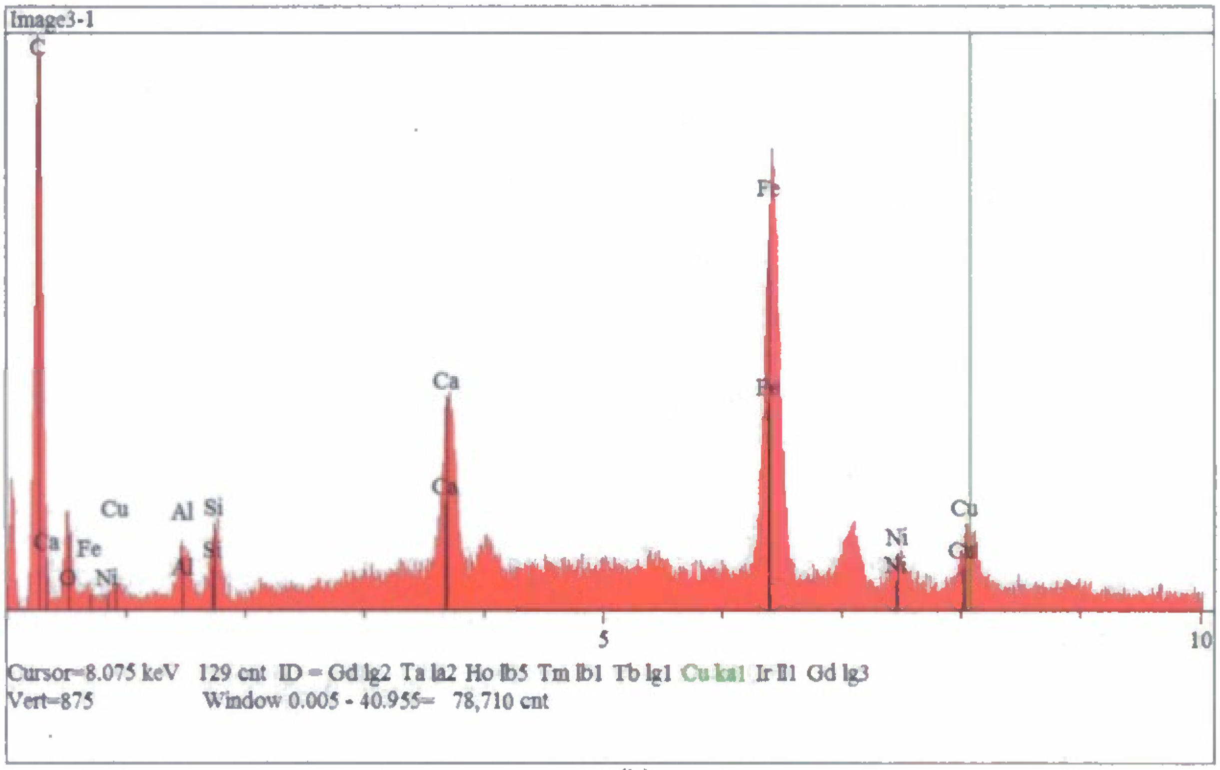
EDAX analysis from the area 2 in (a)

Figure 8
(continued)



(a)
Note the cross mark for EDAX analysis shown in (b)

Figure 9
SEM picture at very low magnification (X500) from as detonation sample A showing impurities



(b)
EDAX analysis from the cross marked area in (a) showing impurities

Figure 9
(continued)

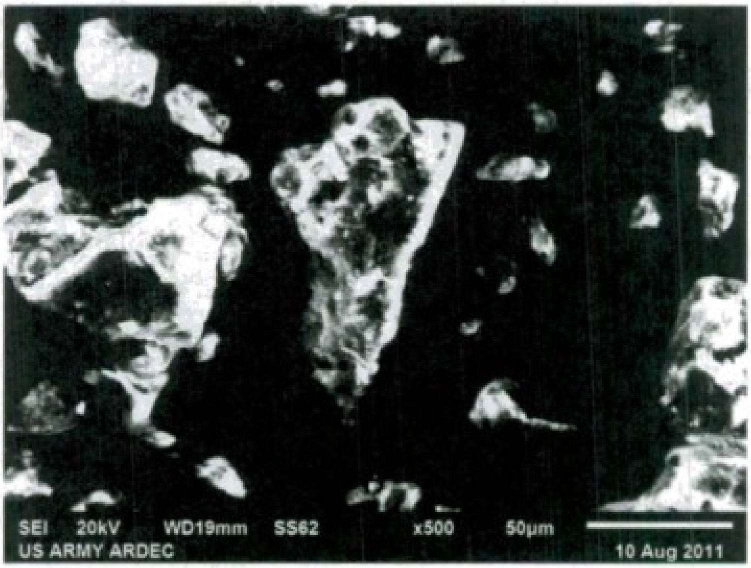


Figure 10
Low magnification (X500) SEM picture obtained from sample B (after purification) showing isolated cluster particles of diamond nanoparticles

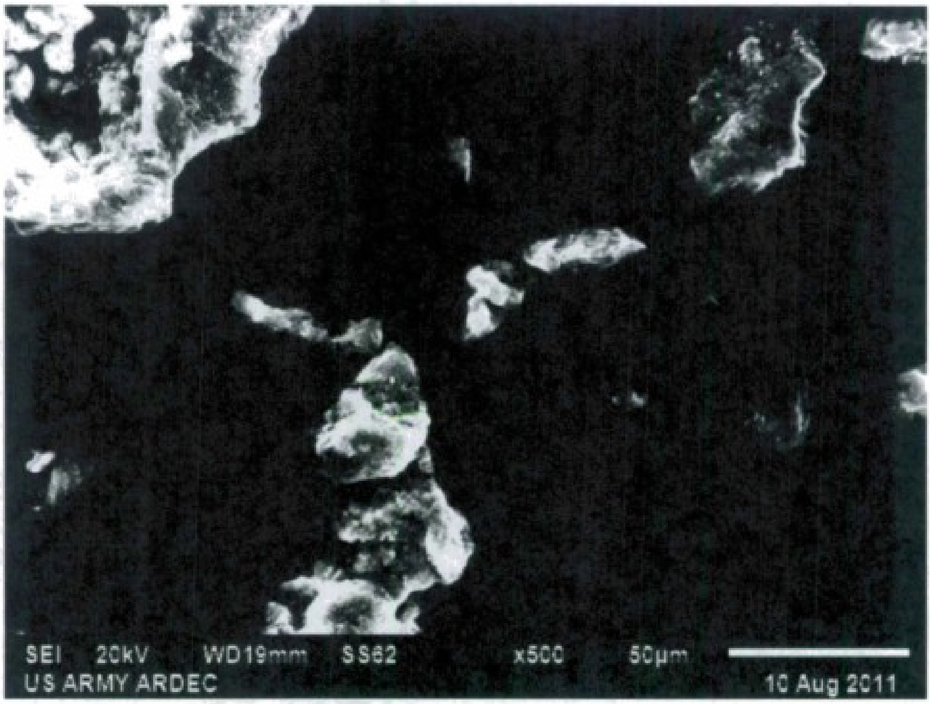


Figure 11
SEM picture taken from different area at same magnification (X500) from sample B

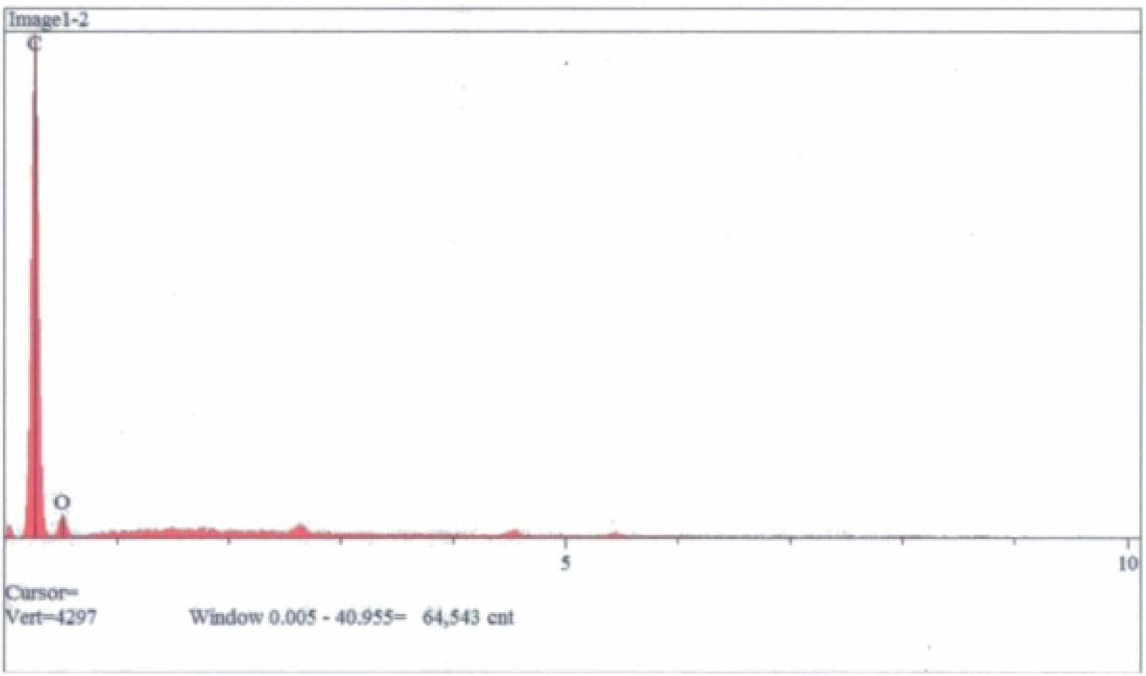


Figure 12
EDAX analysis from the areas shown in figures 10 and 11 showing only the carbon (C) peak due to diamond particles meaning purification process is excellent
(No other peaks due to impurities are present

CONCLUSIONS

The electron microscopy analysis has verified that the purification techniques used by NanoBlox to eliminate contaminants from detonated nanodiamonds was successful.

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