



**HAL**  
open science

## Removal of metal impurities from detonation nanodiamonds by heat treatment with chlorine gas

Kilian Henry, Mélanie Emo, J. Ghanbaja, Brigitte Vigolo, Marc Dubois

### ► To cite this version:

Kilian Henry, Mélanie Emo, J. Ghanbaja, Brigitte Vigolo, Marc Dubois. Removal of metal impurities from detonation nanodiamonds by heat treatment with chlorine gas. Nanodiamonds workshop, Oct 2023, Saclay, France. hal-04280949

**HAL Id: hal-04280949**

**<https://hal.univ-lorraine.fr/hal-04280949>**

Submitted on 11 Nov 2023

**HAL** is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

## Removal of metal impurities from detonation nanodiamonds by heat treatment with chlorine gas

Killian HENRY,<sup>1,2,\*</sup> Mélanie EMO,<sup>1</sup> Jaafar GHANBAJA,<sup>1</sup> Brigitte VIGOLO,<sup>1</sup> Marc DUBOIS,<sup>2</sup>

1 Université de Lorraine, CNRS, Institut Jean Lamour, UMR 7198, allée André Guinier 54000 Nancy, France

2 Université Clermont Auvergne, CNRS, ICCF UMR 6296, 24 av. Blaise Pascal, F-63178 Aubière, France

(\* ) killian.henry@univ-lorraine.fr

Detonation nanodiamonds (DNDs) have the potential to be part of a new class of efficient slow neutrons reflectors.[1-3] However, the synthesis conditions lead to some impurities that are unfavourable for such an application due to their high neutron absorption, e.g. hydrogen, sp<sup>2</sup> C and metal impurities. Thus, ultrapure DNDs are highly required for this application. With the aim of selectively purify DNDs, our approach is based on a combination of gas phase purification treatments: oxidation under air (ODND), chlorination (Cl-DND) and fluorination (F-DND) treatments. The oxidation of DNDs leads to the total combustion of the carbonaceous species, thus concentrating the remaining inorganic impurities which allow their detection (XRD, EDX, FTIR and Raman spectroscopies) and quantification (ICP-MS). Chlorination is used to selectively remove the metal impurities contained in the DNDs samples. The efficiency of the purification treatment is assessed by TGA (Figure 1.A) and FTIR analyses; which is facilitated by applying the selective combustion of the treated DNDs (Figure 1.B). Finally, for the purpose of obtaining ultrapure DND samples, a complementary fluorination treatment is then applied to remove C-H and sp<sup>2</sup> C bonds and impurities which had not been eliminated as volatile chloride in the temperature range (500-900°C) by chlorination. A comparison of the DND samples submitted to these different chemical treatments is visible in Figure 1.C.

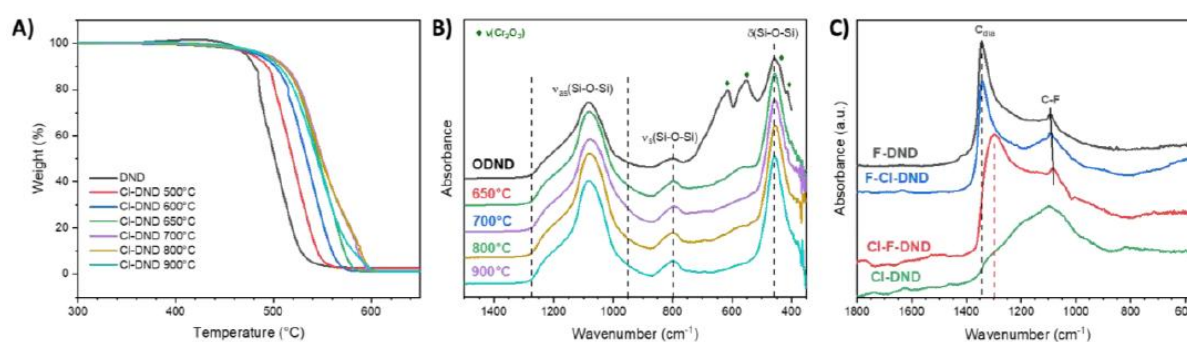


Figure 1. A) Thermogravimetric analysis of DND and Cl-DND samples at different temperatures. B) ATR-FTIR spectra of oxidized Cl-DNDs samples. C) ATR-FTIR spectra of F-DND, Cl-DND, F-Cl-DND and Cl-F-DND samples.

### References

- [1] Nesvizhevsky et al. Carbon, 130 (2018) 799 – 805; [2] Bosak et al. Materials 2020, 13(15), 3337;
- [3] Herraiz et al J. Phys. Chem. C 2020 124 (26), 14229-14236