

## Experiment Report Form

**The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.**

Once completed, the report should be submitted electronically to the User Office via the User Portal:

<https://www.esrf.fr/misapps/SMISWebClient/protected/welcome.do>

### ***Reports supporting requests for additional beam time***

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

### ***Reports on experiments relating to long term projects***

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

### ***Published papers***

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

### **Deadlines for submission of Experimental Reports**

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

### **Instructions for preparing your Report**

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.



	<b>Experiment title:</b> Structural investigation of the interface area between diamonds and metal matrices in high quality diamond cutting and grinding tools	<b>Experiment number:</b> MA-1237
<b>Beamline:</b> ID 13	<b>Date of experiment:</b> from: 02.11.2011 to: 05.11.2011	<b>Date of report:</b> 29.02.2012
<b>Shifts:</b> 9	<b>Local contact(s):</b> Manfred Burghammer	<i>Received at ESRF:</i>
<b>Names and affiliations of applicants (* indicates experimentalists):</b> Andre Steffen <sup>*(1)</sup> , Karin Rüster <sup>*(1)</sup> , Thorsten Brenner <sup>*(1)</sup> , Alexander Nyrow <sup>*(1)</sup> , Michael Paulus <sup>(1)</sup> , Manuel Ferreira <sup>(2)</sup> , Wolfgang Tillmann <sup>(2)</sup> , Metin Tolan <sup>(1)</sup>  (1) TU Dortmund, Fakultät Physik / DELTA, Maria-Goeppert-Mayer-Str.2, 44227 Dortmund (2) TU Dortmund, Institute for Materials Engineering, Leonhard-Euler-Str.2, 44227 Dortmund		

## Report:

Diamond tools are state of the art in grinding and cutting of hard materials as natural stone and concrete [1-3]. The sintered metal matrix serves as a boundary matrix for the embedded diamond. The diamonds are primarily responsible for the grinding process. Therefore the bonding type of the diamonds in the metal matrix is of essential relevance because the interfacial region has to bear up the mechanical forces at each diamond particle [4]. It is of fundamental interest to gain information about this interfacial area e.g. if it consists of metal carbides, solid solutions of carbon in metal, or even graphite. In addition to cobalt which is often used as matrix material nowadays also many alternative materials as iron, copper, chromium and nickel are used as a constituent of the metallic matrix.

At beamline ID13 x-ray diffraction (XRD) studies were performed in order to investigate the interfacial area between diamonds and metal matrices in diamond tools. Using a FreLon 4M camera with 2048 x 2048 pixel of 50x50  $\mu\text{m}^2$  size we were able to gather all Bragg reflection caused by parallel lattice planes with  $d_{hkl} \geq 1.2 \text{ \AA}$ . To achieve high spatial resolution even within the small interfacial area the beamsize was chosen to 125 nm in vertical and 150 nm in horizontal direction. As all measurements were performed at room temperature it was adequate to fix the single diamond grains on a capillary cone end.

Samples	Co mech	Co chem	Cr mech	Cr chem	Fe mech
Quantity of samples	2	5	3	2	2

Table 1: Quantity of measured samples.

In table 1 all investigated samples are listed. Co, Cr, and Fe indicate which matrix material was used. “mech” indicates that the diamond grains were extracted out of the metallic matrix by mechanical treatment, “chem” that chemical treatment in form of nitric acid washing was used. Each counted analysed sample was

investigated by performing grid scans at 5 to 6 different positions on each sample. In figure 1 an optical microscopy image of a diamond grain fixed on a capillary cone end is shown. The XRD scans were performed alongside the orange arrows.

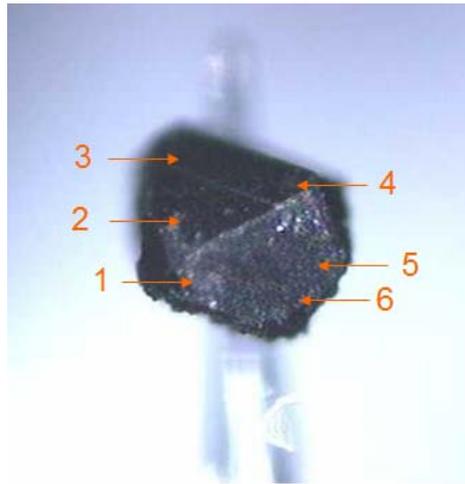


Figure 1: Microscopy image of a diamond fixed on a capillary. The scan positions and directions are shown in orange.

In figure 2 two diffraction patterns, one caused by iron-diamond (left) and one by chromium-diamond (right) interfacial area, are shown exemplarily. This raw data exhibits two interesting features. In the left image a weak but nearly homogenous Debye-Scherrer ring can be identified. This indicates that a significant amount of small polycrystallites is present within the small scattering volume. This may be due to the presence of graphite because the graphitisation of the diamond during the sintering process was observed in former studies. In the right diffraction pattern this Debye-Scherrer ring can not be observed. The dominating scattering signal is caused by strong Bragg reflections that are located within a ring at higher scattering angles. These reflections may be due to chromium carbides as chromium is known to be highreactive in forming carbides. Data analysis is still in progress.

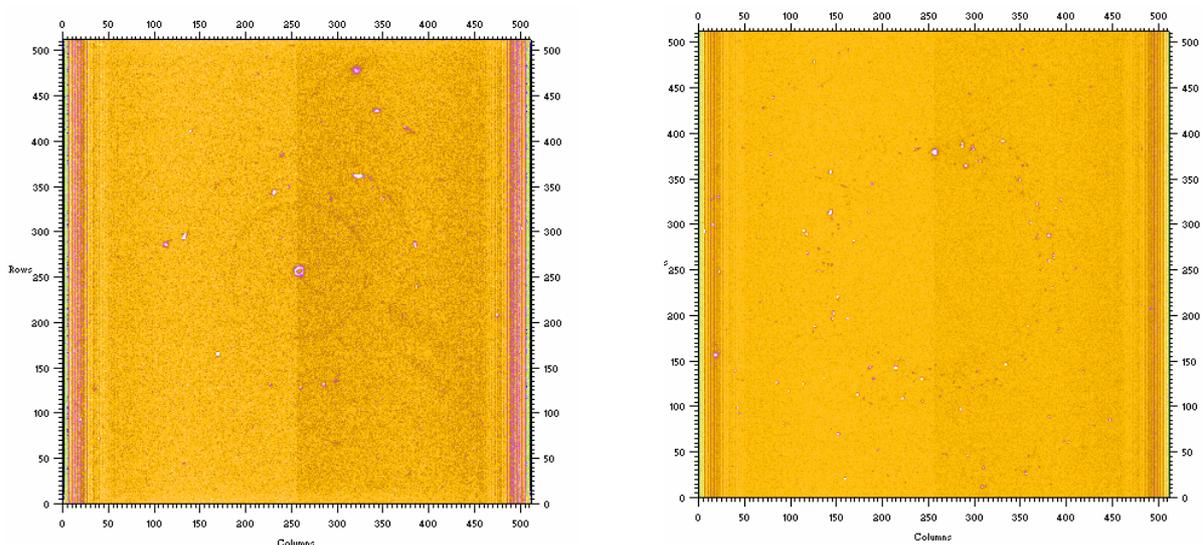


Figure 2: Diffraction pattern of iron diamond (left) and chromium diamond interfacial area (right).

As a conclusion it can be stated that this experiment was successful. We were able to perform XRD analysis within the diamond metal interfacial areas of different samples using three matrix materials (cobalt, iron, and chromium). Therefore, we are optimistic that the data taken at beamline ID13 will reveal the structural information about the composition of the interfacial area between diamond and metal matrices in diamond tools, e.g. if the interfacial areas consist of solid solutions of carbon in metal, pure metal matrices, graphite or metal carbides.

## References

- [1] Y.S. Liao and S.Y. Luo, *J. Materials Science* **28** 1245 (1993).
- [2] A. Molinari *et al.*, *Materials Science and Engineering A* **130** 257 (1990).
- [3] W. Tillmann, *Int. J. Refractory Metals and Hard Materials* **18** 301 (2000).
- [4] A. Romanski, *Powder Metallurgy* **50** 115 (2007).