Experimental report

16/01/2018

Proposal:	**1-02-217**	**Council:**	10/2016

Title: Determination of Bulk Residual Stress Distribution of Superhard SiC-Diamond Samples in Dependence of Heat Treatment and Machining

Research area: Engineering

This proposal is a resubmission of 1-02-187

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Samples: Si-SiC-Diamond ceramics
SiSiC

Instrument	Requested days	Allocated days	From	To
SALSA	4	4	25/01/2017	29/01/2017

Abstract:

Determination of internal stress distribution in the bulk stresses in the bulk of SiSiC ceramics and the change of the distribution by different heat treatments to generate an understanding of the relaxation and as a consequence to generate materials with homogeneous stress fields in the material.
IKTS is engaged in the development of such materials as well as wear resistant superhard SiC-Diamond materials produced without high pressure. Therefore, test pieces also of such materials with well-controlled different thermal history would be prepared for the stress measurement in the bulk.
The result of the measurement could be introduced into finite element models, describing the infiltration of such materials. This would allow better to understand the material formation and behaviour as well as to achieve more reproducible production and more reliable data.

DETERMINATION OF BULK RESIDUAL STRESS DISTRIBUTION OF SUPERHARD SIC-DIAMOND SAMPLES

Scientific background: Ceramic materials quite often consist of different components, which have different thermal expansion coefficients. During cooling from the sintering temperature or during application this results in internal stresses which relax in the near surface area in a different way in comparison to the bulk. It influences the mechanical and wears properties of components. After removal of the surface layer or after heat treatments the stresses can result in buckling or in distortions of the components. Recently high wear resistant super hard silicon carbide bonded diamond materials were developed. These materials can be produced by pressure less infiltration of diamond preforms by liquid silicon. This technology allows producing large scale components for nozzles, bearings and other wear resistant components. It can be produced also as a layered composite [1] (Fig. 1). The material consists of diamond embedded in a SiC-network and some residual Si. This silicon is a main source of internal stresses due to its volume increase during solidification (similar to ice). Hence compressive stress is expected, but cooling down plastic deformation of Si may take place (cf. [2]). Similar situation are found in large seals made from Silicon infiltrated SiC containing besides SiC 10-20 Vol % remaining Silicon, also produced by infiltration. However, in this case the SiC has hexagonal polytypes, while SiC in the SiC bonded diamond material is cubic.

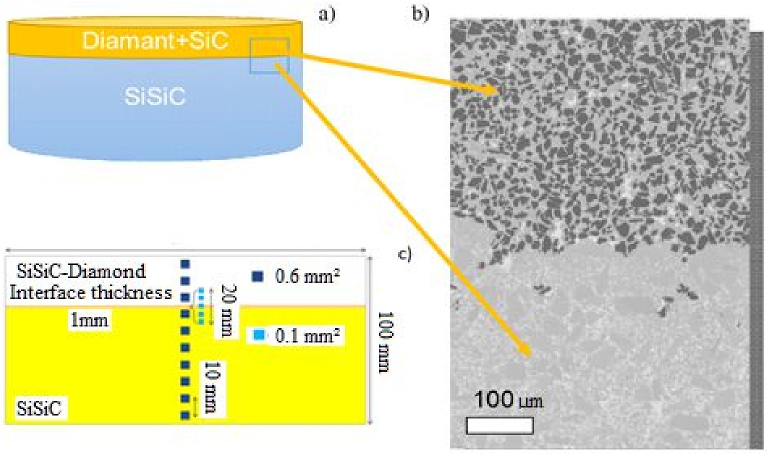


Figure 1: Graded Diamond SiC composite (a) Schematic view of the sample , b) microstructure at the interface, and c) schematic view of the measuring points. Sample dimension: ∅ 30 mm; height 10 mm; thickness of Diamond SiC-layer approximately 3 mm; and mean Diamond grain size 20 µm.

Aim of the measurements: The resulting stress distribution is strongly influenced by the solidification kinetics, which is difficult to measure. Therefore, for these materials it would be very useful to investigate the stress distribution in the bulk to generate an understanding of the formation and relaxation of the stresses and as a consequence to generate materials with homogeneous stress fields in the material. However, the stresses in the components are measured up to now only in the near surface area or by subsequent polishing and measuring by X-ray diffraction or Raman spectroscopy [1]. However, this procedure can result in changes of the stress state due to the material removal. Therefore, it is important to determine the stress in the bulk without influencing the stress state due to preparation for a better understanding and as a consequence an optimal design of such components. Beside this, the data give a fundamental insight in the stress fields generated by reactive infiltration processes. This is of fundamental importance for basic material science.

Results: Using the SALSA instrument at ILL in Grenoble with high spatial resolution of 0,6 and even 0.3 mm different test samples were investigated. The stresses in SiC and graded and ungraded diamond/SiC composites were measured. The stresses in the SiSiC materials are given in Fig. 2. Nearly homogeneous Compressiv stress for Si and low tensile stress for SiC was observed throughout the sample.

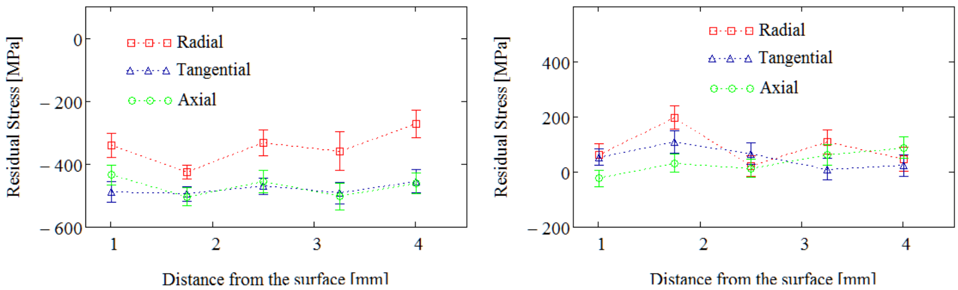
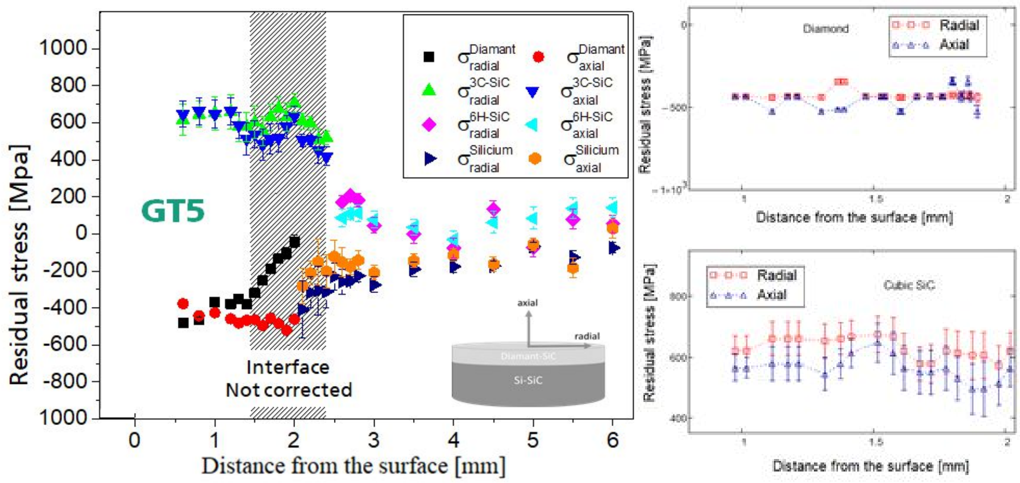


Figure 2: Results of the stress values for a SiSiC sample -left for Si-phase and right for SiC-phase.



- Figure 3: Stress distribution among the constituent phases. Left without and right with interphase corrections for Diamond and cubic SiC-phase. Corrections were done only for Diamond Important for Diamond, since sharp for that phase a sharp border exist, but SiC has no border.

In the silicon carbide bonded diamond materials tensile stresses in the ß- SiC phase (500 MPa) and the diamond show compressive stresses in the same order of magnitude (-450MPa). Similar stresses are also observed in the layered composite – see Fig. 3. It is of interest that the volume averaged stresses over all phases are nearly equal to zero. This result was supported by Raman measurements. However, in the layered sample the stresses of Diamond and SiC-phases don’t compensate completely, here the Si-phase should have compressive stresses of about -100 MPa, which is the consequence of the resulted stress of Diamond and SiC-phases.

Literature:

[1] M. Herrmann, B.Matthey, S. Höhn, I.Kinski, D. Rafaja, A. Michaelis (2012). Diamond-ceramics composites: New materials for a wide range of challenging applications, Journal of the European Ceramic Society, 321915-1923

[2] G.D.Bokuchava, J Schreiber, N Shamsutdinov, M Stalder (2000). Residual stress studies in graded W/Cu materials by neutron diffraction method, Physica B: Condensed Matter 276, 884-885