ESRF	**Experiment title:** Debonding of diamond reinforced metals during thermal cycling.	**Experiment number**: MA-588
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Report:

Materials:

The following materials were measured during the experiment:
Al/CD/60p (60vol.% diamond particle reinforced Al-PRM) monomodal Ø~100µm
CuB/CD/60p (60vol.% diamond particle reinforced Cu+1vol.%B-PRM) monomodal Ø~100µm
Cu/SiC/20m (20vol.% SiC monofilament reinforced Cu-MFRM) Ø~140µm with Ti interface coating

Experiment:

The experiment should show internal stresses and thermal fatigue damage in situ during thermal cycling. The diamond reinforced composites were heated two times to soldering temperature (350°C). The MFRM materials were also cycled twice but up to 550°C adequate to their operating temperature in fusion reactor systems. Simultanous diffraction and tomography at several temperatures were performed during the experiment. For the strain measurements a 2D image plate was used to measure the peak shifts in the principal stress directions relative to matrix measurements. The samples were rotated during acquisition to increase grain orientation statistics and to reduce alignment offsets of the peak positions. Tomography was made with a lower resolution as previously agreed at ~$(2\mu m)^3$/voxel due to setup problems. It was sufficient to show particle delamination and changes in void volume fractions during thermal cycling in the PRM. Also fiber cracks and consolidation voids were visualized in the MFRM.

Current results:

In situ diffraction shows matrix micro stresses between the diamond particles during thermal cycling. In previous investigations on ID19 (MA 300, MA 505), ID15 (MA 57) and FRM2 Garching (ID 555) the effects of reinforcement architectures [1] on micro stress evolution and void kinetics during thermal cycling were shown. In Al/CD/60p, as well as in CuB/CD/60p, a system of isolated particles embedded in a metal matrix can be expected [2]. The particles are moved by an expanding matrix and voids are opened during heating. Tensile matrix stresses are induced in between the particles (fig.1). In situ diffraction shows different stress levels in the two composites. Low stress amplitudes during heating and cooling indicate weak bonding in CuB/CD/60p. Stronger bonding can be concluded in Al/CD/60p causing high stress amplitudes up to ~200MPa. Tomographies of both composites in their initial condition (fig.2), show an initial volume fraction of shrinkage pores and infiltration voids. In Al/CD/60p an initial volume fraction of ~0.5vol.% of voids >5µm was identified. The measured void volume fraction increases from 0.5 to 0.8 vol.% during heating to 350°C. Such voids (~0.7vol.%) identified in CuB/CD/60p are mainly arranged at the interfaces. They originated from processing due to the low wettability of diamond particles with copper during gas pressure

infiltration. Stresses induced during heating increased the void volume fraction up to 10 times of the initial value (from 0.7 to 7 vol.%) by an expanding percolating Cu weakly bonded to the embedded diamond particles. Also fractured diamonds could be observed. Those effects degrade the thermal properties of the CuB/CD/60p composite significantly.

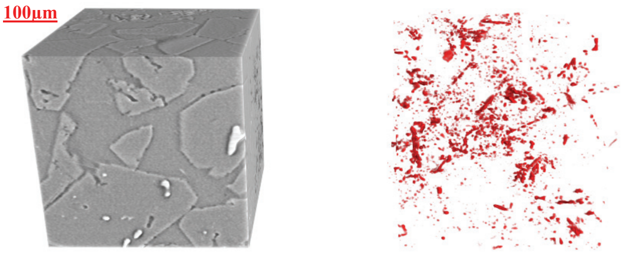

Figure 2a: A (400µm)³ cube of Al/CD/60p as delivered. The voids (~0.5vol.%) are segmented and are shown in red (right).

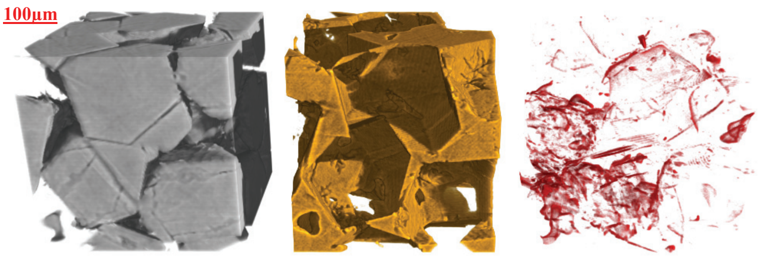

Figure 2b: A (400µm)³ cube of CuB/CD/60p as delivered with the matrix (middle) separated from the diamond particles (left) with an initial void volume fraction (~0.7vol.%) (right), mainly replicating the interfaces.

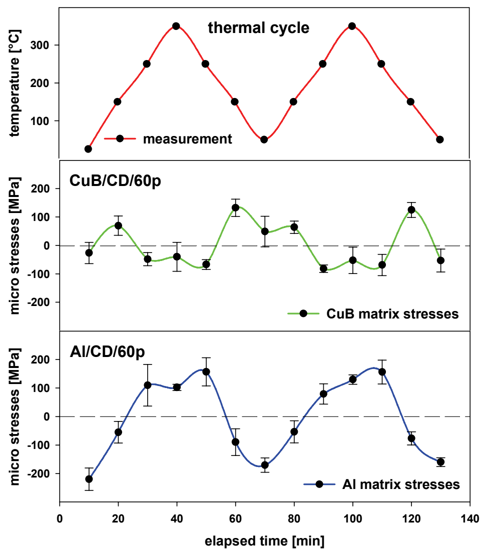

Figure 1: Micro stress evolution in diamond reinforced metals. Tensile stresses are built up during heating and compression during cooling in a system of isolated particles.

Fiber cracks in SiC monofilament reinforced composites are visualized in fig.3. Also voids in between the coated fibers are identified. During heating the fiber fragments get pushed apart by the expanding matrix (fig.4) resulting in the anomalous sample elongation which was also be observed during previous thermal cycling experiments. A dramatic accumulation of thermal fatigue damage under service conditions can be expected with fragmented fibers. The problem arises from the production process.

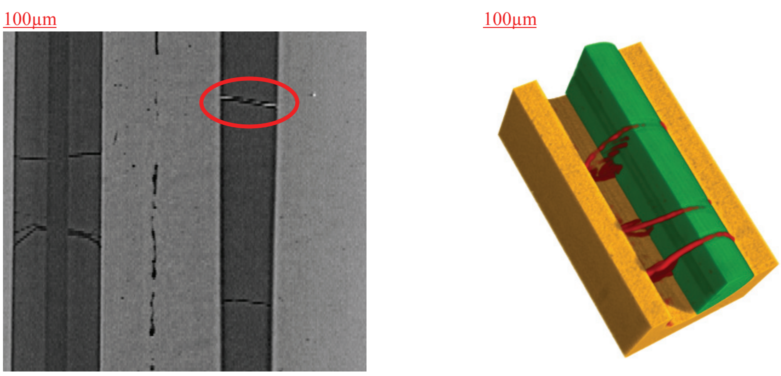

Figure 3: A 2D slice shows fiber cracks in the raw data sets (left). The matrix (bright) is squeezed into the cracks during production. 3D visualization of the fragmented fibers during thermal cycling was possible.

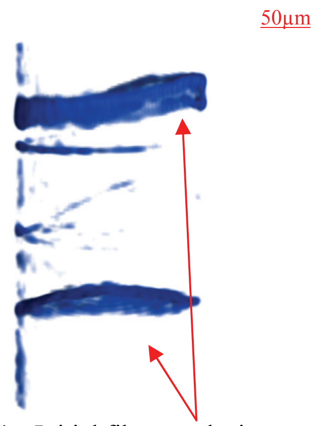

Figure 4a: Initial fiber cracks in MFRM.

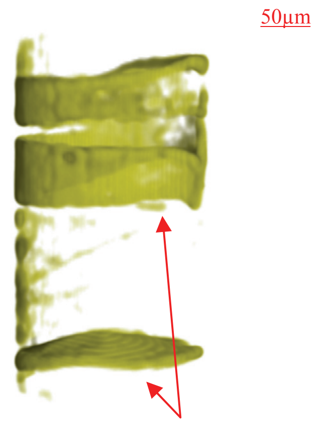

Figure 4b: The same cracks opened after 2 thermal cycles from RT to 550°C.

References:

[1] M. Schöbel, G. Fiedler, H.P. Degischer, W. Altendorfer, S.Vaucher: The effects of different architectures on thermal fatigue in particle reinforced MMC for heat sink applications, Advanced Materials Research, Vol. 59 (2009), pp 177-181.

[2] M. Schöbel, G. Requena, H. Kaminski, H.P. Degischer: Residual stresses and void kinetics in AlSiC MMCs during thermal cycling, Material Science Forum Vol. 571-572 (2008), pp 413-418.