	Experiment title: Residual stress in WC-Co	**Experiment number**: 01-01-611
Beamline: **BM01B**	**Date of experiment**: from: 7.12.02 to: 12.12.02 and from: 9.05.03 to: 10.05.03	**Date of report**: 17.10.03
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Report:

The objective of this experiment is to study the changes of residual stresses in the cermets TiWCN-Co. TiWCN-Co is constituted of a ceramic skeleton: the TiWCN, interpenetrated by the metallic matrix, the cobalt. TiWCN has the typical sodium-chloride cubic structure. Sintering of TiCN and WC powders together with cobalt gives rise to the formation of a characteristic core-rim structure of the TiWCN grains. The rims, which are the remainder of the original milled powder, have exactly the same crystal structure as cores but a slightly larger lattice parameter. Moreover, they show a higher W content part (the inner rim) and a lower W content part (the outer rim). So, we are in presence of a four-phase structure (Fig. 1).
TiCN and Co have very different coefficients of thermal expansion: 8 10^{-6} and 15 10^{-6} [K^{-1}] respectively. This difference should produce high stresses in both phases, at room temperature. The thermal stresses should be relaxed when temperature is increased. Moreover, the core-rim structure should show a distribution of stresses between the three phases.

Experimental results

Residual stresses are assessed by measuring the variations of the lattice parameters of the constituents and by comparing them with a reference supposed unaffected by external or internal stresses:

$$\varepsilon_{hkl}(T) = \frac{d_{hkl}(T) - d_{0,hkl}(T)}{d_{0,hkl}(T)}$$

The thermal expansion must obviously be taken into account. TiWCN composition may vary as a function of temperature so affecting the lattice parameter. We have accurately measured powders extracted from a sintered sample crushed and annealed at 1000 °C. Samples are made of a TiWCN-10vol%Co. They are obtained by spark-cutting rods with 1 mm diameter. Heating is performed under Argon flow in a furnace available on beamline BM01B. Measurements were made by using a wavelength of 0.37 Å for the powders and 0.33 Å for bulk material. In a first experiment, bulk samples seemed to be affected by some macro-stress as the sampled region can be estimated to a depth of 150 µm from the surface. Therefore a second test has been performed on the same sample that can be considered as annealed. The peaks obtained from diffraction of TiWCN did show the superposition of three phases (Fig. 2). The splitting of the peaks is more evident when analyzing less dense planes. By using (220). (311) and (222) diffraction peaks, the lattice parameters of the three TiWCN phases in the cermet were calculated and compared to those of the powders. The variation of the lattice parameter as a function of temperature is shown in Fig. 3. One can observe that the rim parameter deviates considerably from that of powders at high temperature. This deviation cannot be justified by the variation of residual stress and should be attributed to a variation of the chemical composition. It appears that such variation is reversible. The core parameter is much more stable and shows that in reality stresses are very small on the ceramic phase. A good proof that the measurement of the core lattice parameter is correct is given by the measurement of powders with the composition as before sintering (Fig. 3). A more reliable way to assess internal stresses is the measurement of the cobalt lattice parameter. In this case, we don’t have a powder reference, as cobalt is alloyed with tungsten in the cermet. However, the comparison with thermal expansion of pure cobalt gives a good comparison for relative variation. Fig. 4 shows strains and calculated stress in cobalt obtained assuming complete relaxation of residual stress at high temperature. Stress in Co attains 2 GPa in cobalt which corresponds to about 200 MPa in TiWCN.

To summarize, It was possible to detect and measure strains in the three phases composing the epitaxial core-rim structure of TiWCN in a TiWCN-Co cermet for the first time. High temperature chemical composition variations of this material were also observed. Measurement of lattice strains of the cobalt phase give a reliable estimation of residual thermal stress in the composite.

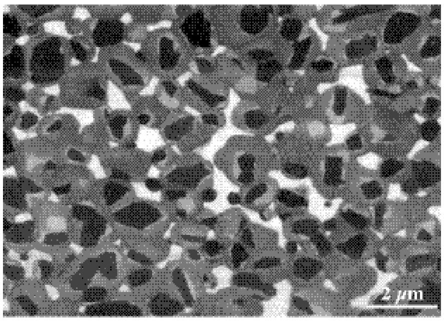

Fig. 1 SEM image of a cermet: the typical core-rim structure is observed. The inner rim appears lighter that the outer rim.

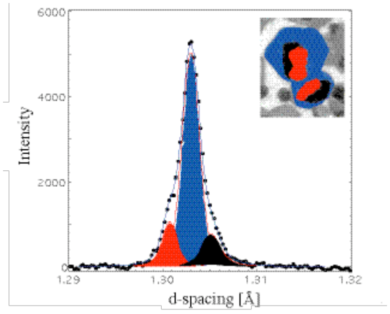

Fig. 2 Peak (311) from TiWCN decomposed into three peaks coming from outer rim (blue), inner rim (black) and core (red).

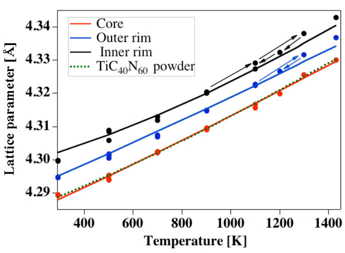

Fig. 3 Variations of lattice parameter of TiWCN in bulk cermet (dots) and in powders (lines). Arrows indicate the sequence of measurements. Dotted line shows the measurement of a $TiC_{40}N_{60}$ powder with same composition as core.

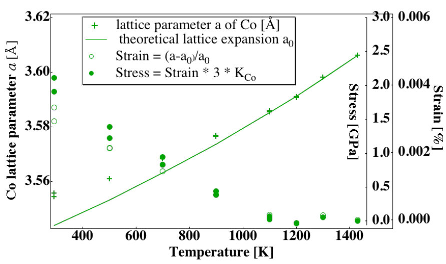

Fig. 4 Measured lattice parameter in cobalt. Strains and stresses are derived from comparison with lattice thermal expansion of pure cobalt. This curve is shifted to match zero stress at high temperature.