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## Measurement of residual thermal stress in WC-Co by neutron diffraction

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#### ABSTRACT

The temperature dependence of residual stresses in a WC-17.8vol.%Co cemented carbide was measured by neutron diffraction. The comparison of the WC lattice parameter within the WC-Co and within stress-free WC reference provides a measurement of lattice elastic strains and, using Hooke's law, stresses. WC is found to be under hydrostatic compressive stresses of about -400 MPa at room temperature, which decrease monotonically with temperature to a near-zero value at 800 °C. Residual stresses in cobalt also decrease with increasing temperature, but show an apparent increase above 800 °C, which is attributed to an increase in lattice parameter due to W dissolution in the Co phase.

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#### 1. Introduction

WC-Co cemented carbides are materials widely used for the manufacturing of cutting tools. They are known for their exceptional combination of toughness and hardness. However, severe cutting conditions implying high temperature and stress produce plastic deformation of the cutting tool tips. Therefore, a better knowledge of the material behaviour at high temperature is needed to improve the quality of the tools. The stress field influencing the microstructure not only derives from the applied load but also from internal stress. As thermal expansion coefficients between WC and Co differ by a factor of three, cooling from the sintering temperature of about 1400 °C to lower temperatures can produce extremely high residual stresses. Measurement of residual strains (stresses) in composites is obtained from the comparison of in situ measured lattice parameters for each phase with a stressfree reference. Diffraction measurements of residual thermal stresses have been performed by different authors [1–6] both by X-rays and neutrons. The high tungsten content considerably limits the penetration depth of X-rays and measurements are sensitive to polishing and annealing. Hence, neutrons are a better choice to measure bulk properties of WC-Co. On the other hand, in WC-Co, due to the high elastic modulus of WC, a very good precision in measurements is required to obtain elastic strains with adequate accuracy. In order to reduce experimental errors in neutron diffraction measurements, differences in positioning and temperature between reference sample and the composite material must be minimum. At room temperature, compressive residual stresses around -500 MPa in WC and tensile stresses around 2000 MPa for cobalt are expected in a WC-18vol.%Co [4]. As temperature is increased, thermal stresses are expected to relax. A curious behaviour was found by [4] where thermal stresses started to increase again by heating above 800 °C. A thermal hysteresis between heating and cooling cycle was also observed. Since strains of the WC lattice are extremely small, it is not clear whether such behaviour should have been attributed to some experimental error. The purpose of this work is to measure the residual stress in both phases of a WC-17.8vol.%Co with high accuracy. We use a particular setup and time-of-flight neutron diffraction in order to achieve maximum precision and temperature reproducibility in the measurements of the composite materials and of the WC and Co references. The changes of residual stresses in the WC and Co phases are measured up to 1156 °C.

#### 2. Materials and experimental methods

WC–Co samples with 17.8vol.% cobalt were prepared by AB Sandvik Coromant (Stockholm, Sweden). This high cobalt grade allows the measurement of both WC and Co diffraction peaks with good intensity. Bars of  $89 \times 19 \times 4.5 \text{ mm}^3$  were produced by a usual sintering process. The SEM micrograph in Fig. 1 shows the general grain morphology. The average WC grain size is 1.8  $\mu$ m.

Neutron diffraction measurements were performed in "time-of-flight" mode using the SMARTS diffraction instrument [7] at the pulsed neutron source at the Lujan Center of the Los Alamos National Laboratory (NM, USA). Detector banks positioned at  $\pm 90^\circ$  angle with respect to the incident beam collect a complete diffraction

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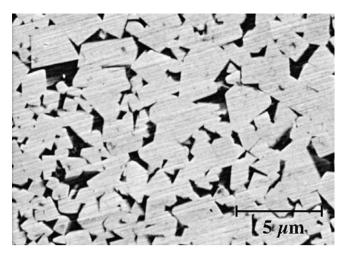
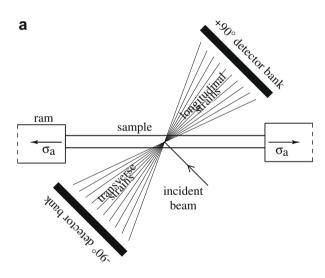
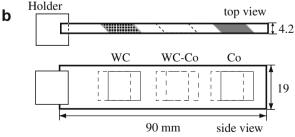


Fig. 1. SEM micrograph of the WC-Co morphology. WC grains are brighter.

pattern from neutrons having a white spectrum produced from a spallation source (Fig. 2a). Diffraction spectra with a d-spacing range between 0.2 and 3 Å are gathered simultaneously for the two banks. The spectra are analysed using the Rietveld refinement method using the GSAS software [8].

We used a particular sample geometry and the possibility offered by SMARTS to translate the specimens inside the furnace (Fig. 2b). Bulk sintered WC–Co samples were spark machined in or-





**Fig. 2.** (a) Schematic diagram of neutron diffraction. The SMARTS system is designed for diffraction under applied load. Crystal planes perpendicular to load axis diffract on the  $-90^{\circ}$  detector bank while those parallel diffract on the opposite direction (b) drawing of the WC–Co frame and windows. In the present experiment, we use the load ram to shift the sample and to expose to radiation different windows in the WC–Co frame. In the WC–Co region, there is no window and the diffracting material is the frame itself. Two independent spectra are collected on both diffraction banks.

der to make two windows at an angle of 45° with respect to the specimen axis to host the WC and the Co references. A slit was used to collimate the beam in the area of interest. With such geometry, the diffracted beam crosses always the same amount of material within the WC–Co support.

In a first experiment, not presented here, we noticed that the use WC powder as a reference may affect the measurement of the lattice parameter due to beam attenuation and consequent shift of the diffraction center of the specimen. In order to obtain a WC reference with a density equal to that found in sintered WC–Co, we etched the cobalt with boiling HCl from WC–Co and inserted the remaining WC 'skeleton' in the WC–Co frame as shown in Fig. 2. A stress-free reference for the cobalt cannot be produced, since in WC–Co, cobalt makes a solid solution with tungsten and carbon with a composition that varies with temperature [4]. Therefore, we used commercially pure cobalt made of 0.5 mm sheets forming a stack inserted in the cobalt reference window.

The sample assembly is mounted inside a vacuum furnace. The temperature of the furnace is controlled by two thermocouples positioned between the measurement windows. The temperature stability recorded during the measurements was better than  $\pm 1$  °C.

The thermal strains in the WC phase are determined for the two lattice parameters a and c of the hexagonal WC lattice with respect to the lattice parameters  $a_0$  and  $c_0$  of the stress-free WC skeleton sample according to equations:

$$\varepsilon_a = \frac{a - a_0}{a_0} \quad \varepsilon_c = \frac{c - c_0}{c_0} \tag{1}$$

Subtracting the reference sample parameters is necessary to offset thermal expansion. Stresses are calculated using WC single crystal elastic constants [9] and the model of Evenschor et al. [10] to calculate stress in the Reuss limit (homogeneous stress) for hexagonal crystals. The following equations are used to convert strain  $\varepsilon$  to stress  $\sigma$  for the hexagonal axes a and c [4]:

$$\sigma_a = 946\varepsilon_a + 259\varepsilon_c \text{ [GPa]}$$

$$\sigma_c = 627\varepsilon_a + 998\varepsilon_c \text{ [GPa]}$$
(2)

The mean hydrostatic stress  $\sigma_h$  in WC can then be calculated according to:

$$\sigma_{WC,h} = \frac{2\sigma_a + \sigma_c}{3} \tag{3}$$

The cobalt has an fcc structure in WC–Co and in our calculations we assume that it is elastically isotropic. The lattice strains in the Co phase result from the sum of mechanical strain in the composite and change in chemical composition of the alloy. In particular, the effect of W and C solid solution on cobalt lattice parameter has been studied by several authors [11–13].

Therefore we can write:

$$\varepsilon_{Co,tot} = \varepsilon_{Co,mech} + \varepsilon_{Co,chem} \tag{4}$$

The mechanical strain should obey the force balance relation:

$$f\sigma_{\text{Co.mech}} + (1 - f)\sigma_{\text{WC.h}} = 0 \tag{5}$$

where *f* is the volume fraction of the Co phase.

#### 3. Results

#### 3.1. Measurement of residual thermal stresses

Two heating-cooling cycles up to 1156 °C were performed. Since the differences between the data collected in the two cycles are small and within the measurement error, the figures presented below correspond to data collected in first cooling and second heating.

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