Quantitative phase and texture-analysis on ceramic-matrix composites using Rietveld texture-analysis

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Ceramic materials can be strengthened and toughened by the inclusion of a second phase in the form of short fibres. Specific anisotropic properties are achieved by fibre alignment along certain directions. To compare the resulting mechanical properties with the expected ones it is fundamental to determine the effective phase quantity along with the orientation distribution-function of both matrix and inclusions. At the present, if the texture of polyphase materials is a difficult task, there is no reliable method for quantitative phase analysis of strongly oriented bulk samples. By Rietveld texture-analysis both features can be analysed at the same time using special measurements affordable at the D20 beamline.

Ceramic materials are increasingly being utilised in structural applications due to their low density, high hardness, thermal stability and resistance to corrosion. However, one drawback of these materials is the occurrence of catastrophic failure due to their intrinsic brittle nature. Single-crystal whiskers or metallic inclusions can be used to increase the fracture toughness of these materials; with respect to the metallic inclusions, the whiskers have the advantage of increasing also the failure strength. In some cases it is desirable to maximise the mechanical properties along certain planes or directions for optimum design of the application. This can be achieved by alignment of the whiskers along the desired directions to obtain textured materials. For research purposes or quality control it is very important to check the texture obtained by the process and the effective quantity and homogeneity of the inclusions. Another important role is played by the residual stresses originating during the material fabrication-process. In most cases these are developed in response to the different thermal expansion coefficients of the matrix and fibres. The nature of the residual stresses (in compression or tension) play a fundamental role in promoting the resulting fracture toughness or so-called crack bridging or crack deflection. The Rietveld method in connection with the measurement of a certain number of spectra collected at different tilting angles seems to be a good procedure to analyse at the same time phase quantity, texture and residual stresses. The first application was to incorporate the harmonic approximation of texture in a least-squares Rietveld analysis but some limits appear, principally concerning the general applicability in connection with sharp texture. Subsequently the texture algorithm of WIMV was included under the general name of Rietveld texture-analysis. Here we try the application of the procedure to a composite material having a matrix of β-Si_3N_4 with SiC whiskers as inclusions. The material was obtained by a special procedure in order to distribute the whiskers randomly in the basal plane of the sample; these whiskers have the (111) plane perpendicular to the fibre direction. In this manner, it is possible to maximise the in-plane fracture properties (strength, elastic modulus and toughness) of the material for applications where a high resistance to bending stresses are required. One purpose of the analysis is to check the angular dispersion of the whiskers from the basal plane and to obtain the residual stresses resulting from the fabrication process. A cubic sample obtained by piling up sheets of the original material was analysed at the D20 beamline. 720 spectra up to 157° in 2θ, were collected with the new position-sensitive detector, corresponding to a 10° * 10° grid on the χ and φ angles and two ω positions of the cradle. The data were analysed by the Rietveld texture-analysis procedure to obtain the texture and the whisker fraction in the sample.

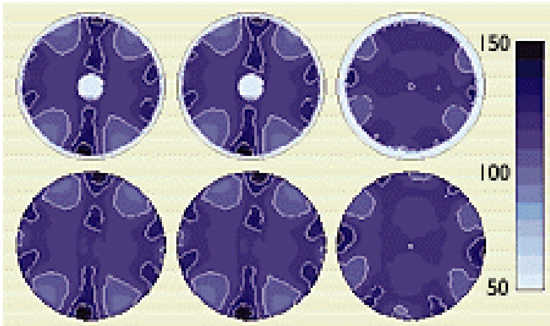


Figure 1: Experimental (upper) and reconstructed (lower), normalised pole figures for SiC. Linear intensity scale multiplied by 100, equal area projection. The pole figures are from left: (111), (-111) and (220).

The fitting results for some of the spectra are presented in Fig. 2. Fig. 1 reports the pole figures as obtained from the Rietveld procedure and the recalculated ones using the WIMV algorithm for the SiC phase; these are labelled respectively experimental and reconstructed pole figures. The sample was positioned in the D20 instrument in a position for which the basal plane containing the whiskers is normal to the pole figure projection and along an ideal line connecting the lower and upper part of Fig. 1. As a result, the maximum polar density for the (111) and (1-11) planes is along that line with a sensible angular dispersion. The silicon nitride matrix was found to be completely random as expected from the fabrication process. The SiC phase fraction obtained by the analysis was 24.2% by volume in good agreement with the expected value and also the crystal-structure refinement on both phases was comparable to existing data. The effective elastic tensor (transversely isotropic) was computed from the phase fractions and orientation distribution function, confirming the expected higher modulus in the basal plane with respect to the normal direction.

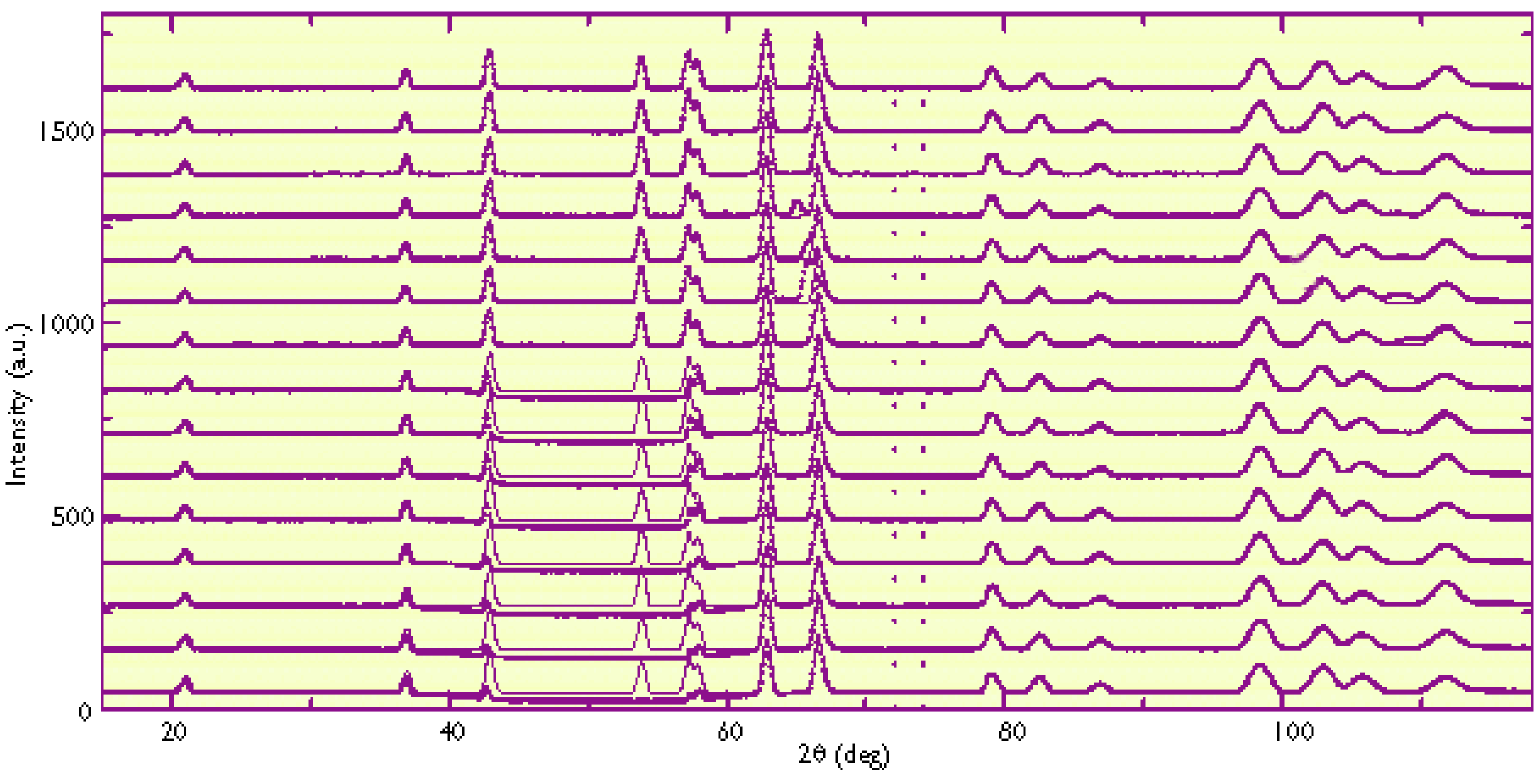
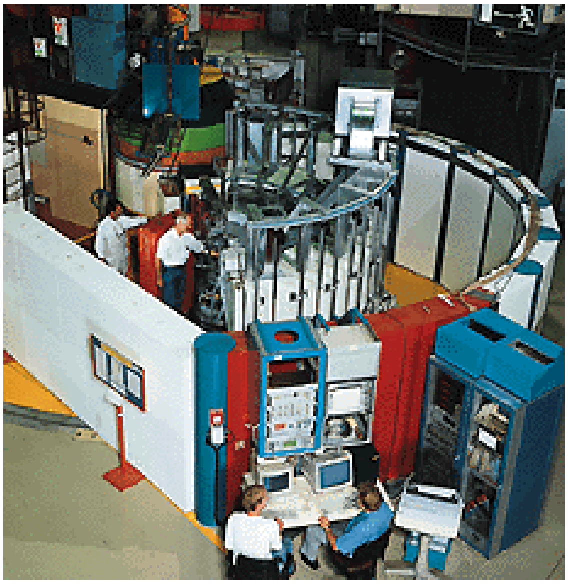


Figure 2: Some of the 720 spectra fitted (thin lines) simultaneously by Rietveld texture-analysis to refine the texture and crystal structure for the silicon nitride sample with SiC whiskers. The low intensity range for the experimental data around 50° correspond to the presence of the Eulerian cradle masking part of the diffracted beam.

The Rietveld method extended to texture analysis applied to data collected at the D20 beamline has enabled a two-phase composite to be analysed for the first time. The results are very encouraging and further work is in progress to obtain the residual stress field from the same data set.

The D20 beamline, thanks to the new position-sensitive detector, permits one to obtain all the required spectra, for a complete analysis, in a time at least two order of magnitude shorter than by traditional techniques, making this procedure of practical use.



View of D20.