ESRF	**Experiment title:** Grinding Damage in Alumina Ceramics	**Experiment number:** HS-1016
Beamline: BM16	**Date of experiment:** from: 4.12.99 to: 11.12.99	**Date of report:** 9.5.00
Shifts: 18	**Local contact(s):** I.Pape	*Received at ESRF:*

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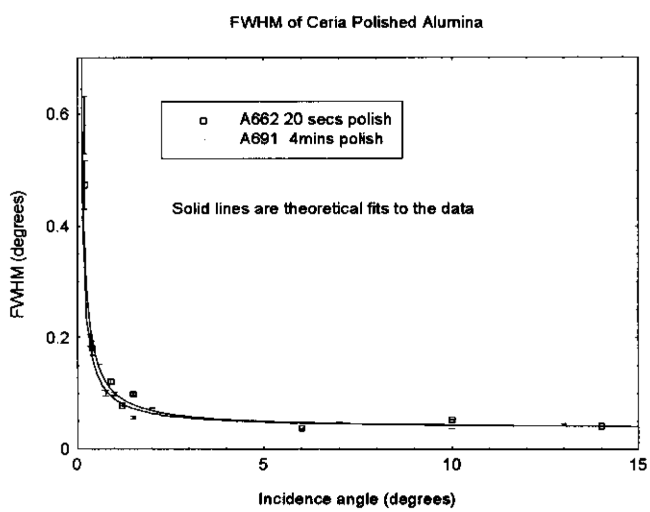
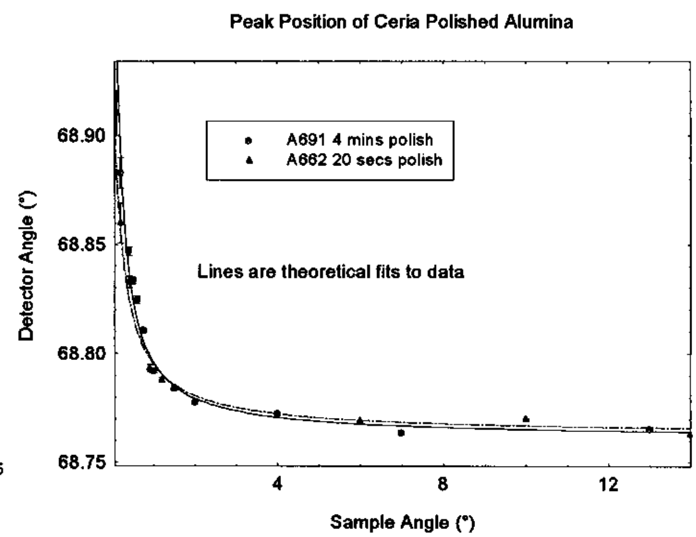
The objectives of this experiment were twofold. The first was to attempt to use very high resolution thin film powder diffraction to measure the state of strain left after polishing of alumina ceramics, while the second was to attempt very high temperature *in-situ* measurements to investigate the origin of the residual room-temperature strain in the sintered materials. We have been extremely successful in achieving the first of these objectives and have performed measurements not only on ceria polished alumina, but have also investigated the effects of annealing and of grit size in diamond polishing of alumina/silicon carbide composites. As a result we had time for only two sintering experiments, in air within the Buhler furnace. The first enabled us to set the parameters for the heating cycle, while the second showed that a rapid cool from 1400°C resulted in very substantial broadening of the peaks in the powder diffraction pattern above that achieved by a slow cool over a period of several hours. We have shown that the experiments are feasible and that the strain can be quantified. The present work forms an excellent basis for a further proposal.

The experiment to determine the sub-surface damage after the polishing process involved precise measurement of the positions and widths of a limited number of diffraction peaks as a function of the (grazing) angle of the incidence beam. During data collection the sample remained stationary and the diffraction pattern was recorded with the high resolution 111 Ge analysers. Initial experiments established that measurements could be made with an incidence angle down to 0.25°, giving a minimum penetration depth of 0.3μm at 8keV. This penetration depth could be tuned up to 25μm with an incidence angle of about 20°.

As very similar variations were observed for prism and pyramidal plane reflections, we concentrated our efforts on the measurement of the 30.0 reflection. For pure alumina samples, horizontal surface ground, diamond lapped and ceria polished for a variety of times up to 10 minutes, we observed no change in either peak position or full width at half height maximum between the very shortest and longest polishing times. In the light of our very extensive study of these samples using grazing incidence diffuse x-ray scattering, optical light microscopy and acoustic wave velocity measurements in which we observed very substantial changes in surface structure and roughness [1], this result was totally unexpected. It indicates that the cracks introduced by the polishing medium are short, propagate ahead of the surface and are not removed. The cracks detected by the acoustic wave measurements are those generated during grinding.





Both peak position (corresponding to macroscopic surface stress) and FWHM (corresponding to variations in stress level) could be fitted well to a simple theoretical model assuming an exponential decay of the stress level below the surface.

On the other hand there were substantially different residual strain states following polishing with 8, 5 and 1μm diamond paste. Although there was no significant shift in the peak position, namely the macroscopic stress in the surface, the full width at half maximum fell, indicating that the random strain distribution was reduced. On addition of SiC to the alumina, the bulk random strains increased, but the surface random strains, which seem to be determined by the polishing process, did not alter. The peak position at high incidence angle decreased as the volume fraction of SiC increased, showing that the bulk of the material was in tension. However, in contrast, the uniform compressive stress in the surface increased with increase of SiC fraction. Annealing of the SiC/alumina nanocomposite that had been ground but not polished, resulted in the amplitude of the random stresses at the surface reducing. We postulate that these random stresses are associated with grain boundaries. The rate of fall of the FWHM with depth decreased commensurately. The compressive strain in the surface increased on annealing. Annealing of polished nanocomposites resulted in no changes in either peak position or FWHM; that is, there were no changes in uniform or random strains. Corresponding macroscopic changes in material strength, previously determined using indentation techniques by Roberts' group in Oxford (who collaborated in the experiments), have been very satisfactorily explained by the x-ray observations.

[1] Evaluation of polishing damage in alumina, I Pape, C W Lawrence, S G Roberts, G A D Briggs, O. Kolosov, A W Hey, C F Paine and B K Tanner, Phil Mag A (in press)