

Raman Spectroscopic Measurements of Molten Ceramic Material at High Temperature

A.G.Kalampounias^{1,2} and G.N.Papatheodorou^{1,2}

¹Foundation for Research and Technology Hellas – Institute of Chemical Engineering and High Temperature Chemical Processes, P.O. Box 1414, GR-26500 Patras, Greece

²Department of Chemical Engineering, University of Patras, GR-26500, Patras, Greece

This article describes a new self-supported technique for containerless processing and study of ceramic materials with Raman spectroscopy. The experimental procedures overcome some of the difficulties in recording Raman spectra of materials at high temperatures due to the relatively weak Raman scattering in comparison with the intense continuous background by thermal emission [1]. The setup as shown in figure 1 consists of a CW Ar⁺ laser for Raman scattering excitation at 90°, a CW CO₂ laser at 10.6 microns/240 Watts in order to heat the samples at the desirable temperatures instead of a conventional furnace. A water-cooled copper device or neutral gas jet are used for supporting the samples. The scattered light is analyzed by a triple monochromator operating at the subtractive mode for better stray light rejection and for sharp cutoff of the exciting laser line at low frequencies and is detected with a two-dimensional charge-coupled device (CCD) detector with the appropriate quantum efficiency in the visible region.

The new high temperature Raman setup has been used to investigate molten ceramic oxide materials formed in the binary system CaO-Al₂O₃, while superheating and undercooling. In order to prepare compositional homogeneous samples we have used the polymerized complex method. This method is described in detail elsewhere [2].

Representative high temperature Raman spectra are shown in figure 2 for the ceramic oxide materials CaAl₂O₄ and Ca₁₂Al₁₄O₃₃ (a eutectic composition). This high temperature Raman system is capable for characterization and study of structural and thermophysical properties of novel materials and has the potential for becoming a powerful tool for in-situ high temperature investigations. Until recently high temperature Raman spectroscopy was impractical because of severe problems of sample degradation during extended exposure to high temperature and signal obliteration due to black body radiation from the sample and furnace.

References

- [1] P.F. McMillan, G.H. Wolf and B.T. Poe, *Chemical Geology*, **96**, (1992), 351-366
- [2] M. Kakihana, *J. Sol-Gel Sci. Technol.* **6**(1), (1996), 7-55

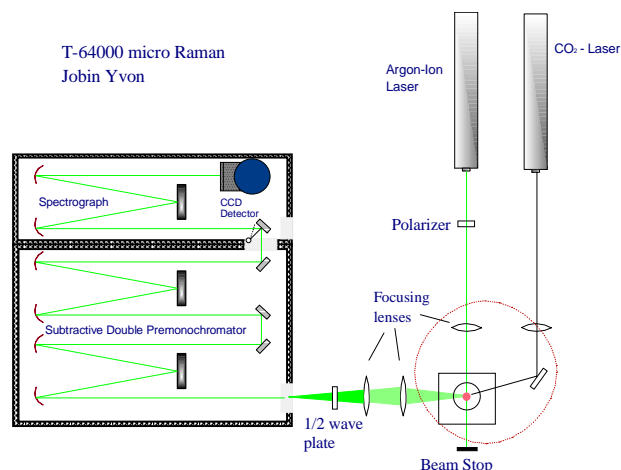


Figure 1. Schematic diagram of the high temperature Raman setup. The setup is based on a CW Ar⁺ laser, a CW CO₂ laser, a water-cooled copper base for self-support technique, collective optics, a triple monochromator and a CCD detector.

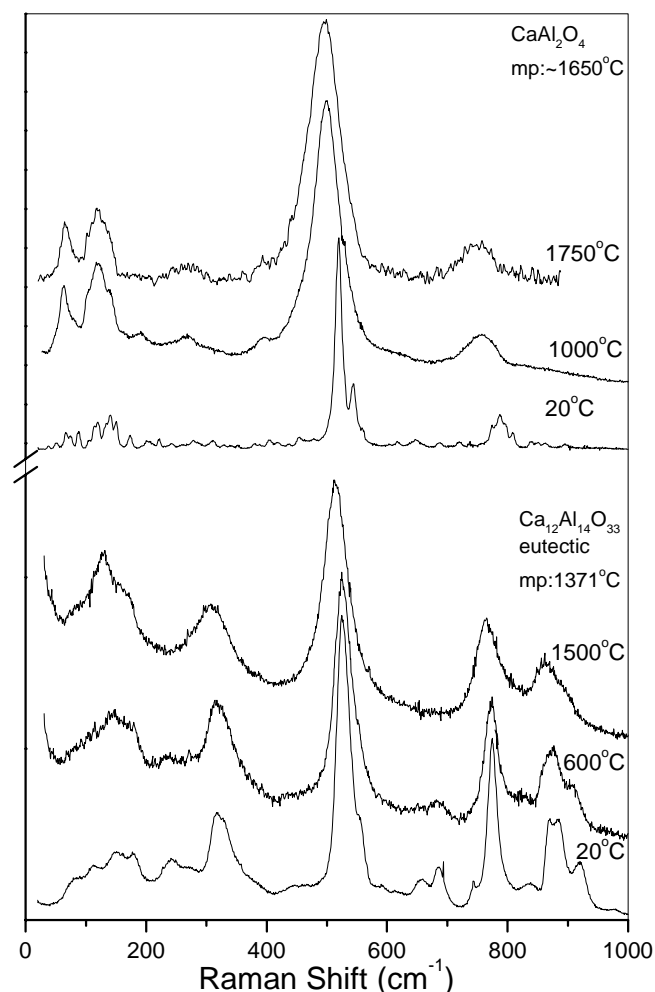


Figure 2. Right-angle Stokes side Raman spectra of Calcium Aluminates as a function of temperature. Excitation line 488 nm.