JOURNAL OF SOLID STATE CHEMISTRY 63, 346–347 (1986)

Answer to "Comment on the Raman Study of the Thermal Transformation of Calcium Hydroxide"

O. CHAIX-PLUCHERY

Institut Laue-Langevin, 156X Centre de Tri, 38042 Grenoble Cédex, France

AND J. C. NIEPCE

Laboratoire de Recherches sur la Réactivité des Solides, Faculté des Sciences Mirande, BP 138, 21004 Dijon Cédex, France

Received November 4, 1985

Despite the arguments used by Dr. Seehra against our interpretations about the Raman study of $Ca(OH)_2$, the second-order Raman spectrum of CaO cannot be observed because of the finely divided state of the material produced by thermal dehydration of the hydroxide. This study is in progress and some new results are announced.

In his comment about our paper (*1*), Dr. Seehra argues against our interpretations of the $Ca(OH)_2$ crystal decomposition. It is worth noting that this paper was published as a brief communication. The experimental results cannot be contested and we have only suggested possible interpretations. These ones were not based on the assumption that CaO is Raman-inactive but on the disappearance of all the hydroxide Raman lines, which allows us to say that the crystal is fully or partially decomposed. As reported by Dr. Seehra, CaO has a second-order Raman spectrum indeed. But in our case, the CaO Raman spectrum cannot be observed because of the finely divided state of the material resulting of the hydroxide dehydration (particle size of about 30–60 Å) (*2*). The Raman effect is mainly a bulk effect and such a polycrystalline sample only gives a large static diffusion (elastic scattering without frequency shift).

Further Raman spectroscopy experiments have been performed and some new results are available.

The use of different exciting lines, i.e., the change of the incident wavelength (4880, 5145, 6471 Å), and the record of both Stokes and anti-Stokes spectra showed that the broad band is not a Raman line. Its frequency shift depends upon the wavelength and it does not appear in the anti-Stokes spectrum. Furthermore, the record of the response curve of the photomultiplier used in the experiments reported in (*1*) revealed that the bandshape was directly affected by this photomultiplier. Spectra registered with another one, of constant response in the spectral domain studied, showed a broad band translated to higher frequencies. This band cannot be attributed to a vibrational mode of H_2O molecules, which was suggested in (*1*). It could be due to a fluorescence transition between two differ-

0022-4596/86 $3.00

ent energy levels generated by the transformations occuring in the solid. It only appears in the spectrum above a given temperature: 130–150°C. Diffraction results show that various modifications take place in the hydroxide structure in this temperature range (*3*, *4*). Nevertheless, at present we are not able to say if a single thermal effect in the hydroxide structure is responsible of such a band or if it is directly or indirectly related to the decomposition reaction of the hydroxide into the oxide. Further experiments are now in progress to precise this point. All the results concerned with the evolution with temperature of the frequency shift, of the intensity and of the width of the band will be published soon in the present journal.

References

1. O. CHAIX-PLUCHERY, D. CIOSMAK, J. C. NIEPCE, AND M. PEYRARD, *J. Solid State Chem.* **53**, 273 (1984).
2. J. C. NIEPCE, thèse d'Etat, Dijon (1976).
3. O. CHAIX-PLUCHERY, J. BOUILLOT, D. CIOSMAK, J. C. NIEPCE, AND F. FREUND, *J. Solid State Chem.* **50**, 247 (1983).
4. O. CHAIX-PLUCHERY, J. BOUILLOT, J. C. NIEPCE, AND J. PANNETIER, *J. Solid State Chem.*, submitted.