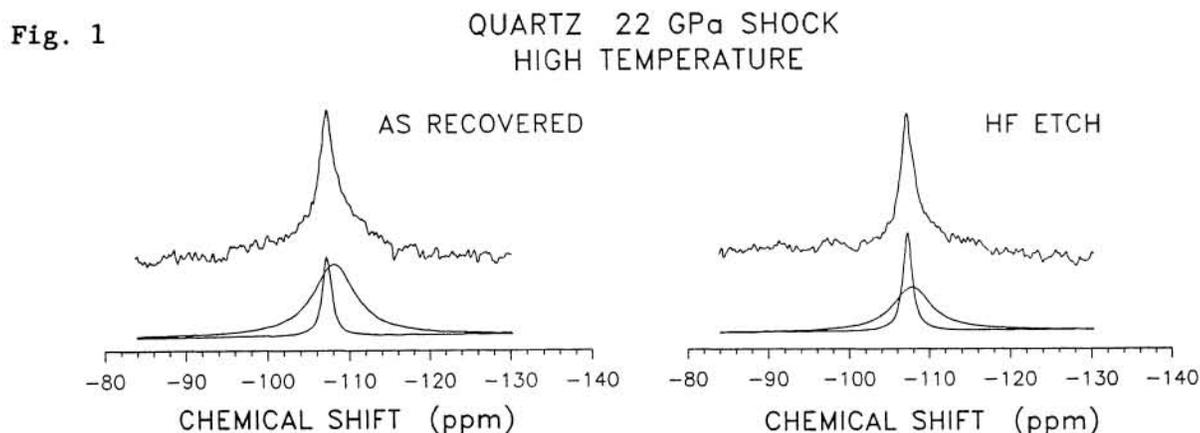


NMR SPECTROSCOPY OF EXPERIMENTALLY SHOCKED SILICATE MINERALS* R. T. Cygan¹, M. B. Boslough¹, and R. J. Kirkpatrick², ¹Sandia National Labs, Albuquerque, NM 87185 and ²University of Illinois, Urbana, IL 61801

Solid state nuclear magnetic resonance (NMR) spectroscopy has been shown to be extremely sensitive to disorder brought about by shock-loading of quartz (1). The technique has distinct advantages for the identification of shocked quartz in bulk samples, as compared to x-ray, electron microscopy, optical, or density methods. We have performed additional shock-loading experiments and NMR analysis of quartz and feldspar samples in an effort to 1) determine reproducibility of previous results, 2) further characterize the NMR spectra with spectral deconvolution, 3) provide additional basis for use of this technique as an empirical "shock barometer", and 4) determine whether more complicated silicates exhibit similar trends.

The series of recovery experiments reported earlier (1) was repeated by shocking synthetic quartz samples at two different initial densities (corresponding to two different shock temperatures at each pressure) to mean peak pressures of 7.5, 16.5, and 22 GPa using the Sandia "Momma Bear" explosive loading fixtures (2). The only substantive difference between these and previous experiments is the manner in which the quartz was prepared prior to shock-loading. The same synthetic quartz was used, however it was annealed at 900°C for 20 hours to minimize the initial defect densities which had previously broadened the (101) x-ray diffraction peak. Unshocked and annealed samples were subjected to extensive characterization by x-ray and NMR techniques. The effect of annealing on the peak widths of NMR spectra was negligible compared to the broadening associated with shock-loading. The ²⁹Si MAS (magic-angle spinning) NMR data from the earlier experiments (1) were fully reproduced in terms of spectral peak widths, with all but one measurement lying within the previously reported error bars.

Decomposition of the measured NMR spectra into two Lorentzian peaks was carried out by way of a six parameter fitting routine (two intensities, two widths, and two positions). The resulting fitting function provides a good fit to the data in all cases for shocked quartz. In Fig. 1, the raw data are shown with the two-peak solution for the 22 GPa high temperature quartz experiment. The two component peaks can be associated with two distinct phases of SiO₂; the narrow peak corresponding to a defective crystalline quartz phase and the broad peak belonging to an amorphous or highly disordered phase (3). Further evidence for this association comes from the reduction of the broad peak after partial dissolution of the recovered powder in HF (Fig. 1), implying



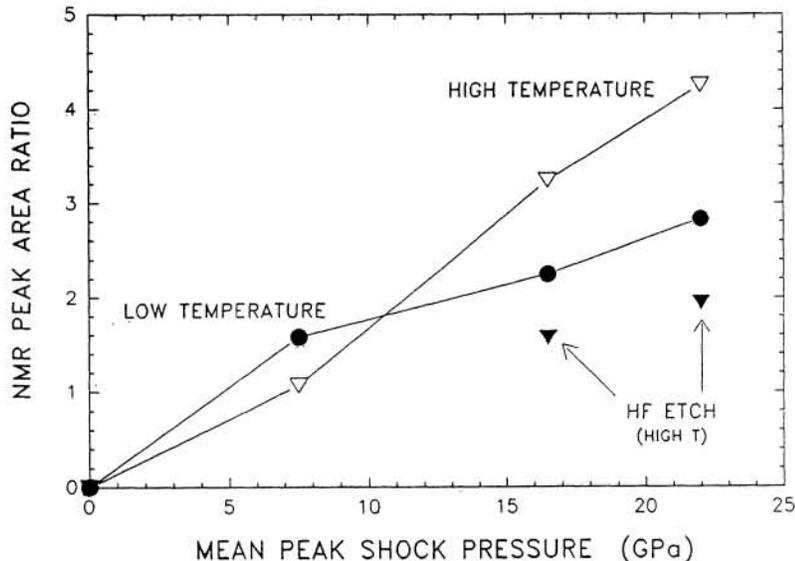
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that the amorphous material is selectively dissolved, as expected. Despite the relatively large amplitude of the NMR signal associated with amorphous silica, there is no x-ray diffraction or optical evidence for glass in the shocked samples. This result implies either that the NMR relaxation time is much longer for silicon nuclei existing in the crystalline phase compared to the amorphous phase, or that the amorphous phase is so finely dispersed within the quartz that it cannot be discriminated optically or with x-ray diffraction techniques. Further NMR experiments using longer recycle times will be performed to answer this question.

The identification of two distinct phases in the NMR spectra provides a strong basis for using NMR spectroscopy of quartz as a shock barometer. The broadening of the overall NMR peak systematically increases as a function of shock pressure, and the deconvolution demonstrates this broadening is due primarily to the increasing contribution of the amorphous component. Since the fraction of silica in the amorphous state is proportional to the ratio of the areas of the component peaks, this ratio (amorphous/crystalline) provides the most useful parameter for evaluating shock pressure dependence (Fig. 2).

In addition to shocking quartz, we carried out experiments on Pueblo Park labradorite for the same three peak shock pressures. NMR spectra were measured for ^{29}Si , ^{27}Al , and ^{23}Na nuclei. Because of the large number of silicon sites in the tetrahedral framework of feldspar, and the higher spin of the other nuclei, the spectra are complicated (consisting of multiple peaks) and are difficult to interpret. There is no evidence from the NMR data for the formation of an amorphous phase in labradorite as a result of the shock-loading.

Fig. 2



- (1) Cygan R. T., Boslough M. B., and Kirkpatrick R. J. (1990) In Proc. Lunar Planet. Sci. Conf. 20th, 451-457.
- (2) Graham R. A. and Webb D. M. (1986) In Shock Waves in Condensed Matter--1985, 831-836.
- (3) Oestrike R., Yang W. H., Kirkpatrick R. J., Hervig R. L., Navrotsky A., and Montez B. (1987) Geochim. Cosmochim. Acta, 51, 2199-2209.

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