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Laser-shock compression of an yttria-doped tetragonal zirconia studied by Raman spectroscopy

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Zirconia is a material of high engineering importance and scientific interest. The most dramatic increase in its industrial applicability has been brought about by the discovery [1] that the tetragonal to monoclinic ($t \rightarrow m$) transformation can be controlled by suitable material processing. Many studies of plasticity and toughening induced by the ($t \rightarrow m$) transformation have also been done in zirconia [2, 3]. A number of studies have also been examined at high pressures and temperatures using Raman spectroscopy [4, 5]. However, there has been little work on shock experiments and the details of the phase transition and equation of state under high-pressures are not well known [6, 7]. In this paper, we studied laser-shock compression of yttria-doped (3 mol%) tetragonal zirconia polycrystals (3Y-TZP) using Raman spectroscopy.

The fundamental light (1064 nm) of a nanosecond Q-switched Nd:YAG (yttrium aluminum garnet) laser (Continuum, Powerlite Plus) was used for shock generation. The pulse width was 10 ns at full width of half maximum. The laser beam of energy 500 mJ was focused onto the target through a multilens array coupled with a normal lens to improve the Gaussian spatial profile to a flat-top profile. The diameter of the focal spot was 1.25 mm. The target assembly, which had plasma-confined geometry, was fabricated with a back-up glass substrate ($100 \times 100 \times 3$ mm^3), an aluminum foil (30 μm thick), a 50 μm thick spacer, the sample and a cover float glass substrate ($100 \times 100 \times 3$ mm^3). The sample used was a 3Y-TZP (50×50 mm^2, 50 μm thick) obtained from Tosoh Corporation. The aluminum foil was bonded to the back-up glass substrate by an epoxy resin (approximately 5 μm thick). By focusing the laser beam onto the aluminum, the generated plasma was confined near the aluminum-glass interface and accelerated the aluminum foil as a flyer. The aluminum flyer collided with the sample and generated a shock-wave. The velocity of the Al flyer was independently measured using an optically recording velocity interferometer system [8, 9]. Peak pressure induced in the sample was determined using the impedance-matching method [10] and the measured flyer speed. In calculations of the impedance-matching method, Hugoniots used were $U_s = 5.15 + 1.37u_p$ for Al [11], $U_s = 7.02 + 2.20u_p$ for 3Y-TZP [6], where U_s is the shock-wave velocity and u_p is particle velocity. The Al flyer velocity was measured to be 0.89 km/s and the peak shock pressure was estimated to be 11.4 GPa. Duration of the shock compression was estimated to be approximately 10 ns from the thickness of the flyer. The samples recovered after shock-compression were examined using Raman spectroscopy. Raman spectra were obtained by a Raman microscope (SPEX, RAMAN-500-UVR). 488 nm light from an Ar-ion laser was focused on the sample with a spot size of 1 μm in order to excite Raman scattering.

Fig. 1 shows Raman spectra of the 3Y-TZP obtained before and after the laser shock compression. Raman peaks (148, 259, 466, and 641 cm^{-1}) due to the tetragonal phase of the 3Y-TZP are observed in the pristine sample. In the shocked 3Y-TZP, Raman peaks (181, 379, 536, 561, and 756 cm^{-1}) due to the monoclinic phase appear in addition to those from the original tetragonal phase. Raman spectra indicate that the ($t \rightarrow m$) transformation is induced by the laser shock compression, whose duration is approximately 10 ns. Fig. 2 shows an expanded spectrum of the shocked 3Y-TZP in the range of 115–220 cm^{-1}.

Two Raman lines of A_g mode of the monoclinic phase are reported to be at 180 and 192 cm^{-1} [12]. However, only the 181 cm^{-1} line is observed in the recovered sample. Siu *et al.* [5] studied Raman spectra of ZrO$_2$ nanograins and reported that the higher frequency peak (at 190 cm^{-1}) shifts towards the lower peak as the grain size decreases and merges to one peak for grain sizes

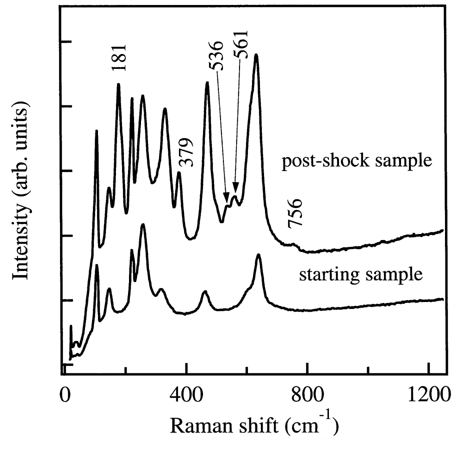

Figure 1 Raman spectra of 3Y-TZP before and after shock compression at 11 GPa.

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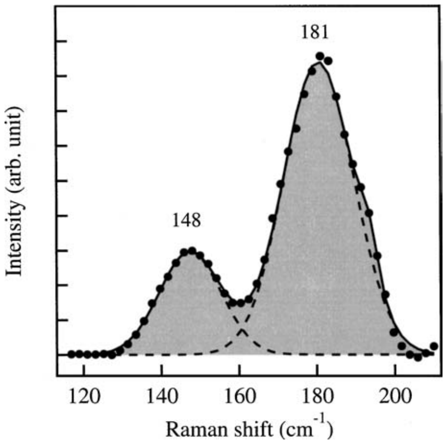

Figure 2 Expanded Raman spectrum of the shocked 3Y-TZP in a range between 115 and 220 cm^{-1}. The obtained spectrum (symbols) is fitted by the sum of two Gaussian functions (solid line) and separated into the peaks which originate from tetragonal and monoclinic ZrO_2 (dashed lines).

less than 12 nm. Therefore, nanograins may be formed by the laser-shock compression.

The fraction (f_m) of the monoclinic phase can be estimated from the intensity ratio (X_m) of Raman lines using the following formula [12]:

$$X_m = \frac{I_{m1} + I_{m2}}{I_{m1} + I_{m2} + I_t},$$

$$f_m = \sqrt{0.19 - \frac{0.13}{(X_m - 1.01)}} - 0.56$$

where I_{m1}, I_{m2} and I_t are intensities of Raman lines at 180, 192, and 148 cm^{-1}, respectively. The Raman intensity ratio was found to be 0.77 and the fraction of the monoclinic phase, which is formed by the shock-induced phase transformation, is estimated to be approximately 30%, which is the maximum value evaluated by the above formula. The ($t \rightarrow m$) transformation induced by the shock compression is also reported from experiments with a powder gun, in which shock duration was of the order of several microseconds [7]. However, the fraction of the monoclinic phase in this laser-shock experiment is much higher than that in the gun experiment: the fraction in the gun experiment is estimated to be 3 and 13% from the Raman spectra shown in [7] at peak pressure of 11 and 32 GPa, respectively. The rapid (nanosecond time scale) change in pressure induced by the laser-shock compression may promote the ($t \rightarrow m$) transformation.

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