Observation of a two-wave shock configuration in zirconium

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The profiles of shock waves with pressure amplitudes from 40 to 280 kbar in zirconium have been measured. Between 93 and 138 kbar, a splitting of the shock wave is observed. This splitting is associated with a first-order phase transition. The phase-transition pressure, equal to the amplitude of the first shock wave, is 62–67 kbar. The transition takes $\approx 0.25\ \mu$s.

As a shock wave propagates through a solid which is undergoing a first-order phase transition, one observes a characteristic splitting of the shock wave,[1)] accompanied by the formation of a two-wave configuration in a certain pressure interval.[1,2] In the shock waves we see the same phase transitions (polymorphic conversions) as occur during static compression. The two-wave shock configuration was not observed in zirconium or titanium until recently although the α–ω phase transition during static compression in these elements was established a long time ago[3] and has been studied quite thoroughly.[4–7] A two–wave shock structure in titanium associated with the α–ω transition was recently detected.[8] We felt it worthwhile to attempt to detect the two-wave structure associated with the α–ω transition in zirconium, especially in view of

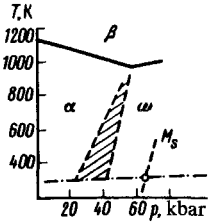

FIG. 1. *T-p* diagram of zirconium. Hatched region—beginning of the α–ω phase transition at static pressures[4–6]; dot-dashed line—Hugoniot adiabat[9]; circle—point of the phase transition and the line M_s from the present study.

the uncertainty and division of opinion regarding the pressure and identification of the zirconium phase transition in shock waves.

Figure 1 shows the *T-p* diagram of zirconium. The ω phase forms at static pressures beginning at 20–40 kbar and persists in a metastable fashion after the pressure is removed.[4–6] During measurements in shock waves, the slope change of the $D(U)$ curve, which occurs only at 260 kbar (Ref. 9), has been attributed to the α–ω transition[9] or a new electronic phase transition[10] of zirconium, rather than to the α–β transition. After the zirconium samples are loaded with shock waves with a pressure amplitude of 120 kbar or higher, a substantial amount of ω phase is observed,[11] implying the α–ω phase transition in the shock waves.

To study the profiles of the shock waves in zirconium we used manganin gauges in the method used previously to study phase transitions in titanium[8] and bismuth.[12] In a first series of experiments we used a small number of samples prepared from an ingot produced by electron-beam melting of zirconium iodide. On an oscilloscope trace obtained at a pressure of 93 kbar (Fig. 2) we found a splitting of the shock wave and the characteristic two-wave configuration which is evidence of a phase transition. The transition pressure, which is equal to the amplitude of the first shock wave (P_1 in Fig. 2), is 67 ± 5 kbar. The blurring of the front of the shock wave, with a nominal pressure of 93 ± 5 kbar (P_2 in Fig. 2), is due to a kinetic phase transition[1] and can be used to determine the duration of the phase transition, which turns out to be ≈ 0.25 μs. We see from Fig. 2 that an elastic precursor[1)] is propagating ahead of the plastic

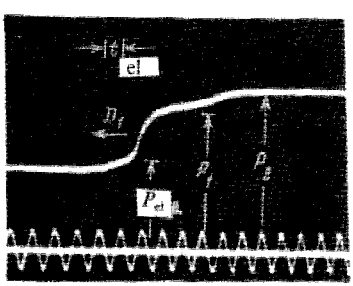

FIG. 2. Oscilloscope trace obtained at a pressure of 93 kbar in zirconium samples produced by electron-beam melting. The frequency of the reference sine wave is 10 MHz.

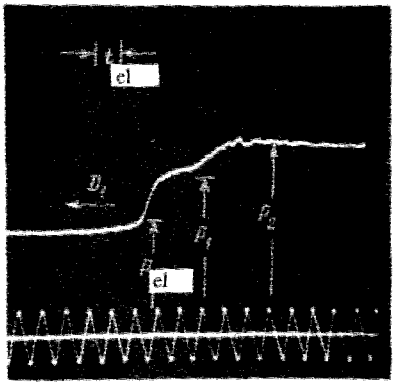

FIG. 3. Oscilloscope trace obtained at a pressure of 96 kbar in zirconium samples produced by vacuum-arc melting. The frequency of the reference sine wave is 10 MHz.

shock wave (P_1 in Fig. 2); this precursor has a gently sloping front, $\approx 0.13\ \mu$s wide, and an amplitude of 14 ± 3 kbar (P_{el} in Fig. 2).

To obtain more information on the phase transition of zirconium in the shock waves we carried out a second series of experiments, in which we used samples made from an ingot produced by vacuum-arc melting of zirconium iodide. These experiments were carried out at pressures near 40, 90, 140, and 280 kbar. At 40 kbar the oscilloscope traces reveal a single-wave configuration of a shock wave with an elastic precursor. At 96 kbar we obtained a trace with a clearly defined two-wave structure (Fig. 3). The pressure of the phase transition is 62 ± 5 kbar[2)] (P_1 in Fig. 3). The front of the second shock wave (P_2 in Fig. 3) is spread out over $\approx 0.25\ \mu$s. On the traces obtained at 135 and 138 kbar we can also see a two-wave configuration, but without the horizontal region on P_1 (Figs. 2 and 3) and with a smooth transition from P_1 to P_2. The width of this transition region is $\approx 0.1\ \mu$s. The profiles of the shock wave at 280 kbar are single-wave profiles without any significant evidence of a phase transition[9] at 260 kbar.

Measurements taken by means of electrical-contact gauges revealed the following parameters for the compression of the zirconium behind the front of the first shock wave (P_1 in Figs. 2a and 3): a wave velocity $D_1 = 4.30 \pm 0.03$ km/s, a mass velocity $U_1 = 0.22 \pm 0.01$ km/s, and a relative compression $\sigma_1 = D_1/(D_1 - U_1) = 1.055$.

In summary, this has been the first successful detection of the splitting of a shock wave due to a phase transition in zirconium, and the parameters of this transition have been determined for the first time. In view of the data on the preservation of the ω phase,[11] we may assume that the P_1–P_2 two-wave configuration observed in these experiments (Figs. 2 and 3) is caused by an α–ω phase transition, and on this basis we have sketched in on the Tp diagram of zirconium in Fig. 1 a line corresponding to the beginning of the α–ω martensitic transition in shock waves (the line M_s in Fig. 1), by analogy with the M_s line on the Tp diagram of titanium.[8]

A question which remains open is the nature of the puzzling phase transition which has been observed at 260 kbar by McQueen *et al.*[9] In contrast with the phase

transition in titanium, which McQueen *et al.* also observed[9] at 175 kbar and which can be identified with the α–ω phase transition, according to a recent study,[7] corresponding experiments on zirconium[5] have shown that at 260 kbar zirconium is still in the ω phase, so that the phase transition at 260 kbar is not the α–ω transition. We should also mention that an x-ray structural analysis of zirconium[6] under static pressure has revealed no phases other than the ω phase at pressures up to 236 kbar maintained for 100 h. In order too explain the phase transition[9] at 260 kbar we evidently need further and more thorough measurements at both dynamic and static pressures.

We wish to thank W. B. Holzapfel for graciously furnishing a report[7] before its publication.

[1)]Here and below, we are talking about "plastic" shock waves, i.e., shock waves with an amplitude above the dynamic yield point, and the splitting of these shock waves due to the phase transition. The elastic precursor causes an additional splitting of shock waves.[1]

[2)]The slight difference between the pressures P_1 in the two series of experiments might be due to the different melting methods and correspondingly different sample purities.

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