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Experimental research on high pressure phase transitions of Mo and Ta*

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Abstract. The high pressure phase transitions of Mo and Ta were investigated experimentally. More melting temperatures were obtained by shock wave experiments. The measured melting temperature at lowest pressure is still much higher than that of DAC experiments. By measurements of sound velocities of Ta in reverse-impact shock wave experiments, a discontinuity of longitudinal sound velocity against shock pressure at ~60 GPa was observed. It may be concluded that a solid-solid phase transition exists.

1 Introduction

Molybdenum (Mo) and Tantalum (Ta) are two of the elements forming the basis of the ultrahigh pressure scale. The equation of state (EOS) of them at high pressure is being used as a calibration standard to the ruby fluorescence in diamond anvil cell (DAC) experiments [1]. Scientific investigations on the phase transitions for transition metals Ta, Mo, W have been extensively conducted experimentally and theoretically, because of their enormous discrepancies in melting curves between laser-heated DAC [2–5] and shock wave (SW) [6,7] methods. As for Mo (as well as Ta and W), several thousand degrees of discrepancies exist in extrapolating from DAC pressures of around 100 GPa to SW pressure of 390 GPa. For Ta, it also belongs to bcc structure at ambient conditions. So far, no bulk phase transitions before melting have been observed by in-situ x-ray diffraction in the DAC experiments up to 174 GPa [4,5,8]. The SW experiments have also confirmed that Ta remains in the bcc phase until shock melting at 300 GPa [9–11]. However, by using transmission electron microscopy, Hsiung and Lassila [12] observed a metastable hexagonal phase in the shear bands of shock-recovered polycrystalline Ta and suggested that a martensitic transformation into the rumpled ω (simple hexagonal) phase occurred in Ta at 45 GPa.

2 Methods

To obtain more melting temperature (T_m) data by SW experiments, two series of experiments have been performed. One is using the technique developed by Tan et al. [13] to measure an additional shock-induced release T_m data of Mo. The other is using porous Mo in SW experiments, with average initial density 9.557 g/cm^3, as the sample material. The details of the experimental technique can be seen in the previous work [14]. The direct reverse-impact configuration together with the velocity interferometer system for any reflector (VISAR) was employed to measure high-pressure sound velocities in shock-loaded Ta. The detailed experimental technique can be seen in previous work [15].

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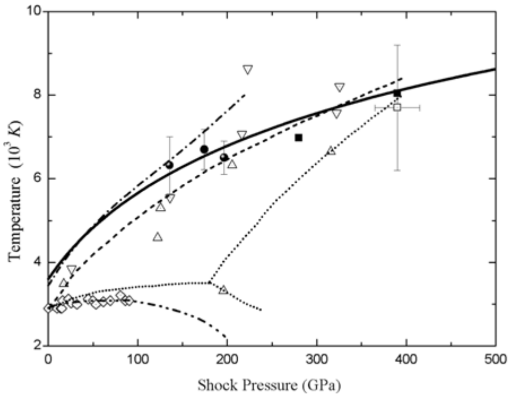

Fig. 1. Melting of Mo. Solid square: our calculated shock-induced equilibrium T_m for solid Mo (at about 390 GPa) and porous Mo (at about 280 GPa). Open diamond: DAC's T_m data [4]; Solid sphere: shock-induced release T_m for solid Mo (this work); Semi-solid sphere: shock-induced release T_m for porous Mo at about 197 GPa (sapphire window) and at about 136 GPa (LiF window); Solid line: our proposed melting curve; Open up- and down-triangles: solid and liquid Mo from *ab initio* MD simulations [20]; Dash-dotted line: generalized pseudo-potential calculations [21]; Dashed line: *ab initio* calculations [22]; Dash-dot-dotted line: semi-empirical model [23]; Short dot lines (except the downwards branch): the two-segment melting curve proposed by [16].

3 Results and discussion

3.1 Melting properties of Mo

With LiF used as the transparent window, we obtained a shock-released melting temperature (174 GPa, 6699 ± 482 K) of Mo. The Hügoniot C results of the porous Mo show that at about 280 GPa, the measured Hügoniot C transformed from longitudinal sound wave C_l to bulk sound wave C_b, and 280 GPa is the shock-induced equilibrium melting pressure. Using the thermodynamic calculations, the obtained T_m for porous Mo is 6978 K (280 GPa). Two T_m data of porous Mo (136 GPa, 6320 ± 682 K) and (197 GPa, 6503 ± 397 K) were measured, which are given in Fig. 1. The new T_m data measured in our work all agree with our melting curve from Lindemann law. However, in the pressure range close to 100 GPa, there still exists large discrepancy in T_m data measured between DAC [2–4, 16, 17] and SW [6] experiments. Furthermore, the dT_m/dP behavior does not significantly change when P-T_m locus evolves across the triple point of *bcc*-*hcp*-liquid, at about 210 GPa, conjectured by Errandonea [16] from Hixson's Hügoniot C measurements [7]. The melting curve in this work is against the two-segment melting curve model proposed by Errandonea [16].

4 Solid-Solid phase transition of Ta

The determined sound velocities are plotted against pressure in Fig. 2. The estimated longitudinal and bulk sound velocities, deduced from Grüneisen EOS and the assumption of $\rho\gamma = \rho_0\gamma_0 =$ constant are also displayed in Fig. 2. In combination with the sound velocity data reported in the literatures [6, 18, 19], we can clearly see that there are two breaks in the plot of sound velocity against shock pressure at ~60 GPa and ~295 GPa. It has been well accepted that the discontinuity at ~295 GPa is resulted from shock-induced melting [6]. Another discontinuity at ~60 GPa, however, is observed for the first time. On the basis of the precision of experimental results, we infer that the discontinuity at ~ 60 GPa likely implies a structural transformation of Ta as the result of Hsiung's shock recovery experiment [12], although it is still impossible to directly determine the structure of the new phase in SW experiments.

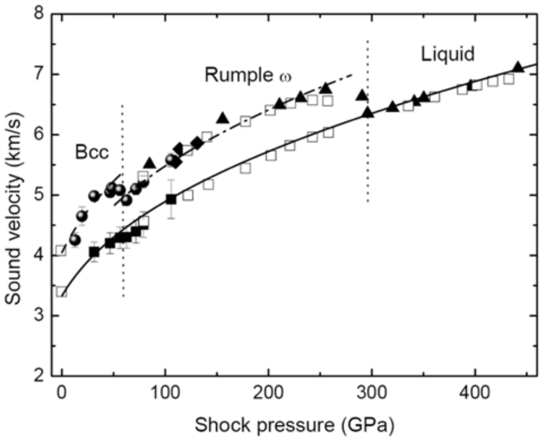

Fig. 2. Sound velocities of Ta. Solid line: the calculated C_b. Dash line: the calculated C_l of bcc Ta. Dash dot line: the calculated C_l of rumpled ω phase. Solid square: C_b in this work. Semi-solid sphere: C_l in this work. Solid triangles: Ref. [6]. Solid diamond: Ref. [19]. Open square: Ref. [18].

5 Conclusions

We investigated the high pressure phase transition properties in Mo and Ta experimentally. The SW experiments were performed to replenish more melting temperature data of Mo. We measured the Hügoniot sound velocity for porous Mo and shock-induced release melting temperature for both solid and porous Mo. The obtained T_m data at the lowest pressure are still much higher than that of the DACs and the overall trend of these T_m data is against the two-segment melting curve model. By accurate measurements of sound velocities of Ta in SW experiments, a discontinuity of longitudinal sound velocity against shock pressure at ~60 GPa was observed, from which it may be concluded that a solid-solid phase transition exists.

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