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Measurement of Shock Residual Temperature in Steel with the Help of Phase Transitions in Zirconium and Titanium

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Abstract. The phase structure of recovered zirconium and titanium samples was studied after loading by plane shocks waves in steel ampoules up to pressure 8-32 GPa. Beginning from some critical pressure residual heating results in full decomposition of the ω -phase. This fact is the basis of evaluation of residual temperature. Obtained values of residual temperature in steel 240 - 500°C in an interval of pressure 23 - 32 GPa turned out to be much higher than values, obtained from temperature calculations usually used for shock waves.

Résumé : On a étudié la composition de phase du zirconium et du titane obtenue sous pression après la mise en charge d'échantillons entre 8 et 32 GPa créée par des ondes de choc planes. A partir d'une pression critique, la température résiduelle dégrade totalement la phase ω . Ce résultat a été pris comme base pour l'estimation de la température résiduelle. Les valeurs de la température résiduelle pour l'acier ont été mesurées entre 240 et 500°C dans la gamme des pressions de 23 à 32 GPa.

At static compression titanium and zirconium transform from hexagonal close-packed modification (α -phase) to hexagonal ω -phase [1]. α - ω transition occurs at static pressure of 2 GPa in titanium and 2.2 GPa in zirconium. The phase transformations in Ti and Zr also occur during shock loading. The breaks on Hugoniot are detected at 17.5 GPa in titanium and at 26 GPa in zirconium [2]. To which transformations these breaks correspond is not clarified till now. As a rule, the values of phase transition pressure at static and dynamic loading are close. Shock transformations in Ti and Zr, on the contrary, take place at considerably superior pressures than pressures of equilibrium.

The hysteresis of transformations in Ti and Zr is so great that the high pressure phases recover after removal of both static and dynamic pressures that allows to carry out studies of a phase structure and its dependence on conditions of loading.

As samples we used Zr and Ti disks with 16 mm in diameter and 1 mm in width. The samples were put in the recovered ampoules made of stainless steel X18H10T (24 mm in diameter and 16 mm in height) and loaded by plane shock waves with pressure amplitudes from 7 to 32 GPa.

The experiments were carried out at initial temperatures (T_i) 0 and -180°C. The phase structure was determined using x-ray diffraction. Results of experiments are in fig. 1.

From compression and unloading conditions, P - T diagram of Zr and Ti and temperature stability of phases at atmospheric pressure [3] we may conclude that the curve of ω -phase concentration in the recovered samples as a function of the pressure $\eta_\omega(P)$ should have three parts: raising, beginning at a phase transition pressure, plateau (or maximum) and consequent lowering. This lowering is caused by the residual heating effect on ω -phase; it is known that at atmospheric pressure this phase is not stable and decomposes during heating, being transformed into stable α -modification [4,5].

Figure 1: Concentration of the ω -phase in recovered samples $\eta_{\omega}(P)$ after shock loading at different initial temperatures.

The appearance of ω -phase above 8 GPa agrees well with the data on the shock profile measurements [6,7] and can be the direct confirmation that shock front splitting, registered in [6,7], is really associated with the α - ω transition. Preliminary cooling of the recovered ampoule to -180°C and relative lowering of residual temperatures result in increase of an interval of pressure, at which the ω -phase is recovered.

The difference between dependencies $\eta_{\omega}(P)$ for titanium and zirconium is only in the value of pressure at which the ω -phase appears and disappears in the recovered samples. This difference agrees with the data from the works [6,7], where with the help of manganese gauges the greater pressure of phase transition in titanium, than in zirconium, was fixed; and the works [4,5], where smaller temperature stability of the titanium ω -phase, recovered after static compression, was marked in comparison with the zirconium ω -phase.

Shock temperatures and residual temperatures after unloading, as a rule, are calculated with the help of equations of state, obtained from shock experiments data. Residual temperature at zero pressure is estimated with use of equation

$$\frac{T}{T_0} = \left(\frac{V_0}{V} \right)^{\Gamma} \tag{1}$$

where T and V refer to the compressed state, T_0 and V_0 - to unloaded. Residual temperatures in stainless steel X18H10T calculated with the help of this equation are the following: at source Hugoniot pressure of 20 GPa $T_0=45^{\circ}\text{C}$, at $P=30$ GPa $T_0=90^{\circ}\text{C}$. For these evaluations we used the equation of state with constant Gruneisen coefficient Γ .

By similar way it is possible to calculate temperatures in zirconium and titanium. We may also take into account additional heating in shock wave, caused by the phase α - ω transition. Considering this heating, residual temperature in Zr, calculated with the formula (1) at 30 GPa is 90°C . We did not consider the reverse ω - α phase transition since it is known, that the high pressure ω -phase recovers in the samples.

Thus, according to this calculations, residual temperatures after loading to $P \leq 30$ GPa in materials of ampoules and samples should not exceed $\sim 100^{\circ}\text{C}$. Obviously, that is not so, because in reality the ω -phase does not decompose at such temperature effect. Below we shall try, on the basis of obtained $\eta_{\omega}(P)$ -dependencies and the data on temperature stability of the

ω -phases, to restore actual residual temperatures, realized under conditions of our experiments.

From the analysis of $\eta_\omega(P)$ dependencies for zirconium and titanium it is clear that, at first, with increase of pressure η_ω grows, reaches a maximum, then decreases and since some critical value P^* becomes equal to zero. As behind the shock front ω -phase concentration cannot decrease with increase of pressure, we judge, that this decrease is caused by increase of residual temperature in the steel ampoule and the sample itself.

So, above some pressure P^* , and consequently beginning with some T_0 , the high pressure phase in recovered samples completely decomposes, and its concentration becomes zero. It means that residual temperature also reaches some critical value T_0^* . Thus, by study of high pressure phase decomposition kinetics in laboratory conditions, it is possible to simulate the decomposition process in real conditions of sample cooling after loading and unloading.

During the experiments at $T_i=0^\circ\text{C}$ the ω -phase in zirconium, loaded up to 24.5 GPa and higher, did not recover. In experiments at $T_i=-180^\circ\text{C}$ the concentration of the ω -phase becomes zero since greater pressure - ~32 GPa. Similar picture was observed in the experiments with titanium.

The kinetics of transformations in the solid state at constant temperature is frequently described by the Avrami equation

$$\zeta = 1 - \exp(-kt^n); \quad k = k_0 \exp\left(-\frac{E}{RT}\right) \quad (2)$$

where ζ is the part of reacted substance; R - the gas constant; t - time, k_0 , n , E - parameters, determined from experiment.

The question on the mechanism of ω - α -transformation is not clarified yet, there is particularly an assumption that it is isothermal martensite. Nevertheless, despite of the real mechanism, the Avrami equation, intended for the description of transformations with nucleation and growth, describes satisfactorily our experimental data.

To determine the parameters in the kinetic equation of the ω -phase decomposition, the samples, containing a significant amount (~80-90 %) of the ω -phase were subjected to isothermal annealing of different duration at temperatures of 220 and 300°C for zirconium and 150 and 200°C for titanium.

Further let us extend the values of Avrami equation parameters, obtained in isothermal conditions, to the nonisothermal case. Let the steel cylinder (the ampoule with a sample) cool down on air from temperature T_0 . We accept air temperature to be equal to zero. Temperature in the center of the cylinder thus varies as:

$$T = T_0 \exp(-bt). \quad (3)$$

The parameter " b " depends on the geometrical size of the cylinder. From a combination of the equations (2) and (3) it is possible to obtain a kinetics of ω - α transformation during continuous cooling of the sample. The results of calculations, that give a picture of temperature decomposition of the ω -phase, are shown in fig. 2. Here we consider that at T_0 a sample and an ampoule are in thermal equilibrium.

From these calculations it follows that after some time (~100 sec) from a beginning of cooling of the sample, consisting of the ω -phase, the amount of the ω -phase in it is stabilized because of reaction velocity decrease, caused by lowering of temperature, or, if the initial temperature is great, the ω -phase decomposes completely. In fig. 3 the dependencies of final concentration of the ω -phase in samples on initial temperature, i.e. on residual temperature after unloading are shown.

Figure 2: Time dependence of the ω -phase concentration in the sample during cooling from various temperatures (calculations).

Figure 3: Dependence of the final ω -phase concentration in the sample after cooling on residual temperature T_0 .

Thus, if residual temperature in the ampoule is 320°C and higher, we obtain a pure α -phase in the recovered zirconium sample, if the temperature is ~270° and lower - we see as much of the ω -phase in the sample, as it was immediately after unloading, before cooling.

Now, taking into account obtained kinetic calculations, it is possible to make the following deductions from the analysis of curves $\eta_\omega(P)$ (fig. 1). Firstly, let us consider the experiments with zirconium at $T_i=0$ (fig. 1a). Beginning from $P=24.5$ GPa the ω -phase in the samples does not recover. Therefore, residual temperature in an ampoule (sample) after

loading by this pressure is $\sim 320^\circ\text{C}$. In experiments at $T_i = -180^\circ\text{C}$ the ω -phase does not recover since $P=32$ GPa. We put this pressure in correspondence with the residual temperature of $320 + 180 = 500^\circ\text{C}$ (here we assume that thermal capacity of steel in the considered interval of temperatures is constant).

Similar comparison was also done for titanium. We put pressure of 23 GPa (fig. 1b) in correspondence with the temperature of full decomposition of the ω -phase 240°C (fig. 3). Pressure of 29 GPa - with the temperature $240 + 180 = 420^\circ$.

Having four curves $\eta_\omega(P)$ we may obtain four residual temperatures in a steel ampoule (fig. 4).

The obtained values of residual temperature are much higher than values obtained by calculations (less than 90° at pressure up to 30 GPa). It is necessary to mark that the discrepancy in values of calculated and experimental residual temperatures in stainless steel of a similar composition was also found in [8] with use of the optical method.

Figure 4: Pressure dependence of residual temperature in stainless steel.

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