RESEARCH ARTICLE

Application of Millisecond Fast Compression Technology in Materials Science

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Abstract: The research on the effect of millisecond fast compression on the structure and properties of materials is still in its infancy, it is of great scientific significance to improve the experimental technology of rapid compression and to continuously and deeply carry out the research on the high-pressure physical properties of materials and the preparation of new materials under rapid compression. The paper introduces 4 kinds of millisecond-level rapid compression experimental techniques, briefly describes the application of rapid compression technology in materials science, including preparing amorphous materials, measuring Grüneisen parameters and W-J parameters, and studying phase transition kinetics.
Keywords: Rapid compression; pressure-induced solidification; amorphous material; Grüneisen parameter; W-J parameter

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1 Introduction

In the past hundred years, high-pressure physics has achieved fruitful results, fully revealing that pressure, like temperature and composition, is an independent thermodynamic basic parameter that determines the structure, state, and properties of a material system. The traditional high-pressure experiment science is divided into static high pressure and dynamic high pressure. In the static high pressure experiment, the pressure loading time is generally on the order of seconds to minutes, usually after reaching a certain pressure value, keeping the pressure constant for scientific research, so the pressure loading process is often ignored. In dynamic high pressure experiments, the pressure loading time is in the order of nanoseconds to microseconds, which is usually regarded as an adiabatic process or a quasi-isentropic process. At present, the pressure loading process between static high pressure and dynamic high pressure, especially the research on the pressure time in the millisecond level, is still very few, and it is a vast world that needs to be further explored. Many interesting and unexplored physical phenomena lie between the loading time scales of static high pressure and dynamic high pressure. A unified understanding of static and dynamic compression across related time and space scales is considered to be one of the top five challenges of compression science in the future. It is of great scientific significance and application prospects to carry out the research on the compression process between static high pressure and dynamic high pressure loading time scale. The thesis will introduce the rapid compression experiment technology developed in recent years, combined with the work of this research group in the rapid compression, briefly describe the application of the rapid compression experiment technology in materials science.

2 Fast compression experiment technology

The literature reports on fast compression technology mainly include the following four aspects.

(1) In the 1970s, Boehler et al. [1,2] in the United States based on the thermodynamic relationship of the equation of state, proposed a fast compression process to simulate adiabatic conditions, and calculated the Grüneisen parameter by measuring the rate of change of the material temperature (T) with the increase in pressure [3]. The method to realize the rapid compression process is: immerse the sample in the liquid pressure transmission medium in the piston cylinder high pressure mold, quickly open the con-

trol valve, so that the liquid in the reservoir quickly pushes the piston, and the piston transmits the force through the liquid pressure transmission medium To the sample. In order to make the measurement result as close as possible to the instantaneous rate of pressure change with temperature, Boehler et al. used a small boost range (ie 0.1 GPa) in the rapid boost experiment, and the boost time was about 100 ms, and the corresponding boost rate It is about 1 GPa/s, and the highest pressure is close to 5.0 GPa after using a solid pressure transmission medium [8].

(2) Since the 1990s, people have combined rapid compression technology with on-line measurement technologies such as synchrotron radiation X-ray diffraction, infrared absorption spectroscopy and nuclear magnetic resonance, and carried out research on kinetic problems in protein, biological macromolecule and liquid crystal phase transition [4,9].

Its working principle is: open the control valve to connect the high-pressure gas (or liquid) with the working chamber, make the pressure in the chamber rise instantaneously, and transfer the force to the sample device to realize rapid pressurization. The rapid compression process occurs within a few milliseconds. The device has small volume and is easy to carry, but its sample chamber is small and the pressure is low. Due to the limitation of medium phase transition, the pressure of such experiments is usually within 1.5Gpa, and the maximum pressurization rate is less than 140Gpa / s [6].

(3) Since 2001, our research group has carried out experimental research on fast compression technology, and designed and developed a large-scale fast compression experimental device [9]. The technical route is: by opening the connecting valve, the high-pressure oil of the accumulator is injected into the master cylinder, so that the oil pressure of the master cylinder rises rapidly to the level of the accumulator. In this process, the master cylinder pushes the piston to apply the force to the high-pressure die. The typical measurement results are as follows: the pressure of the main oil cylinder rises from 1GPa to 30gpa within 20ms and remains stable for a long time; A pressurization rate of up to 500 GPA / s and a pressurization amplitude of 10 GPa are generated on the sample by pressing the anvil on the cemented carbide plane. The volume of the sample cavity is in the order of cubic millimeter to cubic centimeter. This is the first large-scale rapid compression equipment built in the world. The pressure loading time of the device is in the millisecond level, which is in the blank area between the traditional static high pressure and dynamic high pressure loading time scales.

(4) In 2007, Evans et al. Of the United States reported a dynamic diamond anvil (ddac) loading system. It uses piezoelectric ceramics to exert force on Diamond cavity. It is a reciprocating small cavity rapid compression device. The pressurization time is determined by the waveform generator controlling piezoelectric ceramics. In principle, the device can realize the pressurization rate of 500Gpa/s and higher pressure, which is convenient to carry out the study of phase transition dynamics in combination with the on-line detection means with high time resolution. Therefore, it has attracted extensive attention of researchers in China and the world [5].

3 application of rapid compression technology in material science research

3.1 application of rapid compression technology in the preparation of amorphous materials

There are many preparation methods of amorphous materials, most of which can be classified as melt quenching process. However, the size of amorphous materials formed by this quenching method is limited by the thermal conductivity of materials. For the preparation of amorphous materials under high pressure, it is also reported in the literature. For example, Mishima et al. Carried out compression experiments with diamond anvil and found that there is pressure-induced amorphous phase transition, but the high-pressure amorphous phase can not be recovered under normal pressure. Wang Wenkui put forward the high-pressure exposure principle of metastable phase, that is, high-pressure quenching technology. It is found that the critical cooling rate of melt solidification into amorphous under high pressure is reduced, which is conducive to the preparation of bulk amorphous materials; However, since the melting point of the material usually increases with the increase of pressure, the method needs to heat the sample to a higher temperature, and the cooling rate of the sample under high pressure is limited by the pressure medium. Yang et al. Prepared zirconium based amorphous alloys by impact high pressure technology. The amorphous materials obtained by this method are considered to be formed after the molten samples are rapidly depressurized and cooled. In short, whether static high-pressure quenching technology or impact high-pressure technology, the main role in the formation of amorphous is the temperature drop, and it is still unable to avoid the limitation of thermal conductivity on the size of amorphous materials.

In 2005, this research group first proposed the use of rapid compression technology to prepare amorphous and other metastable materials, referred to as melt rapid pressure-induced solidification method [10]. For most substances, the melting point (T_m) increases with the pressure (p) within a certain pressure range. Pressurizing the melt

of these substances may obtain subcooling under high pressure, thereby making it solidification. In principle, as long as the pressing speed is fast enough and the degree of subcooling obtained is deep enough, metastable materials such as amorphous materials can be prepared.

The important thing is: during the rapid pressurization process, the solidification of the melt does not depend on the change of temperature, but the change of pressure. The surface and interior of the sample are solidified under pressure at the same temperature at the same time, so the heat conduction has no effect on the solidification process. Which fundamentally solves the problem that the size of amorphous and other metastable materials is limited by thermal conductivity. This is different in principle from the melt quenching method, which is very beneficial to the preparation of bulk amorphous materials of larger size. This research group has used millisecond-level rapid compression technology to prepare a variety of amorphous materials. Two typical examples are listed below. We have prepared bulk amorphous sulfur (referred to as fast-pressed amorphous sulfur) by using the melt rapid pressure-induced solidification method. Compared with the amorphous sulfur prepared by the quenching method and the pressure-induced amorphization method, the fast-pressed amorphous sulfur has high thermal stability. The amorphous sulfur prepared by the quenching method will crystallize rapidly at room temperature. Even at the glass transition temperature Tg (233~258K, which is related to the thermodynamic process of preparing the sample), part of the amorphous sulfur will slowly crystallize into α-phase crystals. Another method to prepare amorphous sulfur is the crystal pressure-induced amorphization method reported by Sanloup et al., which destroys the crystal structure by applying high pressure. They used synchrotron radiation X-ray diffraction (WAXS) technology at 40~175K and 50~100GPa at low temperature and high pressure. However, this kind of amorphous sulfur is easy to crystallize even at low temperature and high pressure. Fast-pressed amorphous sulfur has high thermal stability, and the distinguishable crystallization peak appears after being placed at room temperature higher than Tg for 75 minutes, so there is enough time to study amorphous sulfur at room temperature, such as stretching at room temperature Induce amorphous sulfur crystallization and so on. Further research shows that the exothermic melting of amorphous sulfur is accompanied by obvious volume expansion, and its melting temperature rises with the increase of pressure, but it does not conform to the Clapeyron equation of the usual first-order phase transition. In addition, we also convert the liquid sulfur before and after the lambda transition (liquid-liquid phase transition at 432K under normal pressure) at different temperatures into two types of amorphous sulfur through rapid pressure-induced solidification. The color and transparency of crystalline sulfur are similar to those of liquid sulfur before and after lambda transformation, and both have high thermal stability and high elasticity, which provides favorable conditions for further research on the lambda transformation mechanism of liquid sulfur. In short, the success of the rapid pressure-induced solidification method to prepare amorphous sulfur has brought a series of new discoveries and understandings to the study of amorphous sulfur phase transition.

Polyetheretherketone (PEEK) is a polymer material with very low thermal conductivity. At present, the size of the PEEK amorphous coating obtained by plasma spraying, flame spraying and other technologies is less than 1mm. The micro-area X-ray diffraction (XRD) technique was used to analyze the structure of the two kinds of PEEK block samples at different depths in the axial profile. The results show that the PEEK block material prepared by the quench method has an amorphous structure on the surface layer less than 1mm, while the rapid compression solidification method The prepared PEEK block is completely amorphous from the surface to the center. The importance of this result is that it clearly shows that the critical size of the amorphous sample prepared by the rapid pressure-induced solidification method can far exceed the quench method. In the past, the critical size was used to evaluate the amorphous forming ability of the material and it was not suitable for the rapid pressure-induced solidification process. . In addition, the performance test results show that the PEEK amorphous bulk material prepared by the rapid pressure-induced solidification method has high tensile strength and excellent impact resistance and wear resistance. In short, the rapid compression technology can not only prepare larger-size amorphous bulk materials, but also bring new properties to amorphous materials, and even obtain brand-new amorphous materials.

3.2 Application of rapid compression technology in phase change research

Regarding the phase change problem caused by pressure, static high-pressure experiments or theoretical calculations usually only give the equilibrium phase diagram. For many simple material systems, the phase change is considered to be completed in a very short time, so basically it is difficult to observe the phase change process without paying attention. A small number of studies on the dynamics of phase transitions under static high pressure are to observe the relationship between phase transition time and temperature by maintaining a certain pressure,

rather than to study the dynamics of pressure changes with time. In our previous research work, we found that there are a large number of material systems, and the phase transition process is in the microsecond, millisecond, or even longer, especially for materials with different cluster structures, multi-component alloy systems, and macromolecules. For complex systems, the phase transition time can be quite long. The study of the kinetics of the phase transition of these material systems will surely provide an important reference for understanding the phase transition process of more materials. Through the rapid pressurization device with continuously adjustable pressurization rate, the use of different loading processes in microseconds, milliseconds or seconds, combined with different heating processes under high pressure, can carry out pressure-induced phase transition kinetics studies of different material systems.

3.3 Application of rapid pressurization technology in physical parameter measurement

The Grüneisen parameter (γ) is an important parameter of condensed matter. It connects the elastic properties of matter with the thermodynamic properties, and provides an important way to study the high temperature and high pressure effects of various thermodynamic quantities and to establish the high pressure state equation of matter. Due to the importance of the Grüneisen parameter of matter under high pressure in the fields of condensed matter physics, thermodynamics, and geophysics, many theoretical calculations and experimental measurements have been carried out around it for a long time.

The experimental measurement of Grüneisen parameters usually uses the relationship between Grüneisen parameters and some measurable thermodynamic variables established by the partial derivative relationship of the Grüneisen equation of state, and the Grüneisen parameters are obtained by measuring these thermodynamic variables. In 11977, Boehler et al. gave an approximate expression $\gamma=(Ks/T)(\Delta T/\Delta p)s$ based on the thermodynamic relationship of Grüneisen parameters (where Ks is the elastic modulus of the insulator, and the subscript "s" represents the adiabatic process), A rapid pressurization process is proposed to simulate adiabatic conditions, and the Grüneisen parameter γ is calculated by measuring the temperature change ΔT when the pressure rises Δp. In order to make the temperature-to-pressure increase ratio (ΔT/Δp) as close as possible to its instantaneous rate of change, the used boost amplitude Δp needs to be as small as possible, but this also causes the uncertainty of the measurement results to increase.

The research group used rapid compression technology to carry out the experimental measurement of Grüneisen parameters under high pressure, and made two improvements to the measurement method of Boehler et al. [7]:

(1) Use a large-scale rapid pressurization method to replace the past small-scale pressurization, and then use the differential median theorem to calculate the temperature change rate at the midpoint pressure before and after the pressurization, which can increase the effective number of measured data and reduce it Uncertainty can also get the accurate rate of change of temperature to pressure at the midpoint pressure.

(2) Through the analysis of the actual temperature drop curve in the high pressure holding stage after pressurization, it is found that it contains the heat loss information caused by heat conduction. As long as the temperature drop curve is differentiated, the temperature drop rate of the system at different temperatures can be obtained. Considering that the same heat dissipation behavior also exists during the pressurization process, the calculated cooling rate can be added to the heating curve of the pressurization process. It is equivalent to retrieving and making up the temperature drop caused by non-adiabatic factors during the pressurization process to obtain an ideal adiabatic process temperature rise curve.

We used an improved method to successively measure the Grüneisen parameters of Nacl, Cu, Fe, Pb and other substances and their relationship with pressure, and the pressure range reached 0~7.5GPa, and discussed the influence of temperature on the Grüneisen parameters of Nacl and AL. Similarly, our research group proposed a method to directly measure the W-J parameter (R) using fast compression technology. The W-J equation of state is a "volume equation" with pressure and temperature as independent variables proposed by Wu Qiang and Jing Fuqian. It complements the Grüneisen equation of state and is an extension and improvement of the entire region's equation of state theory. As an important parameter in the W-J equation of state, the W-J parameter is related to the macroscopic and microscopic properties. Its determination is of great significance to the research and application of the thermodynamic properties of substances and the high-temperature and high-pressure equation of state.

We choose NaCl as the sample, and measure the temperature and pressure changes of the sample in situ during the rapid compression process. According to the slope of the temperature drop curve of the sample itself under constant high pressure, the temperature drop information caused by heat conduction is obtained, and the temperature value during the pressurization process is corrected to make the measured value more consistent with the result under adiabatic compression in principle. Adopting

a large-scale pressurization technique combined with the differential median value theorem, the change rate of the temperature at the midpoint pressure with the pressure is obtained, which avoids the influence of the pressurization range on the accuracy of the measurement result of the change rate. According to the relational expression R=(p/T)(∂T/∂p), find the W-J parameter R. The whole process does not introduce any empirical parameters, which avoids the uncertainty caused by other parameters in principle. Using the above experimental method, we also studied the W-J parameters of graphite, Ta, Mo, Fe, Cu, Pb and other materials under high pressure.

In addition, we also use millisecond-level rapid compression technology to establish an experimental method for measuring the isentropic compression line of matter: firstly, it is demonstrated that the maximum pressure and the corrected maximum temperature during the rapid compression process correspond to the isentropic compression line. At the same point, multiple sets of temperature and pressure maximum values are measured through rapid compression processes of different amplitudes, and multiple points are determined and fitted to obtain an isentropic compression line. Using this method, the isentropic compression lines of Mo at pressures below 12.8 GPa and Ta and graphite at pressures below 5 GPa are obtained.

4 Conclusion

At present, the millisecond-level rapid compression experiment technology is still in its infancy, and there is still a lot of research work to be carried out in the development of a large-cavity press with a continuously adjustable pressurization rate and a combination with high-time-resolution online measurement methods. In addition, many interesting topics need to be studied in depth, such as the preparation of new metastable materials (such as new component binary or even elementary metal amorphous materials), the study of metastable phase transition and its phase transition kinetics, including temperature, pressure, and time. The PTTT phase diagram of multiple dimensions, the study of the mechanical properties of materials in the special high strain rate range between static high pressure and dynamic high pressure, etc. These issues are related to the fields of earth and planetary science, material science, energy science, etc., and have broad application prospects in aviation, aerospace, weapons, and functional materials. In short, it is of great scientific significance to improve the technical level of millisecond-level rapid compression experiments and continue to carry out research on material properties and synthesis of new materials under rapid compression conditions, and is an important research field worthy of vigorous development.

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