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ARTICLE

Sedimentation Process and Phase Transition of Solids Under High Gravity

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Abstract: The recent progress and future prospects for ultra-centrifugal sedimentation in solids are described, mainly involving equipment, miscible systems and compounds. Almost 90% ultracentrifugation experiments were performed on the 1st and 2nd high-temperature ultracentrifuge which is typically operated at temperatures below 500 °C under the maximum centrifugal acceleration up to 10^6 g. The strong gravitational and temperature fields induce atomic-scale graded structure, grain growth and refinement, and voids accumulation caused by the atomic sedimentation in miscible systems. New structures, properties and substances are produced in some compounds. A new cantilever high-temperature ultracentrifuge with a test temperature up to 1200 °C is under construction at Zhejiang University, making it possible to simulate the composition, microstructure and property evolution of superalloys in the operating environment of aircraft engines.

Key words: high gravity; sedimentation; diffusion; superalloy

Known as "HiGee" since the 1970s[1], high gravity technique has been widely applied in chemistry, biology and energy for separation, rectification, absorption, etc[2–6], as shown in Fig. 1a. As a process intensification technique, it enhances gas-liquid-solid mass transfer by rotating the centrifuge at a high speed to generate centrifugal force which is 10−1000 times higher than gravity[7]. On this basis, solidification casting under high gravity has been derived[8–10]. Except solidification, in recent years, there are more researches on the sedimentation process in solids under the strong gravitational field (10^5 – 10^6 g level) and high temperature field, as shown in Fig. 1b. When the solid specimen rotates in the high-temperature ultracentrifuge, it will be affected by the high gravity (referring to centrifugal force). As the high gravity increases from the axis to the outer edge, graded structure appears in the ultra-centrifuged solid specimen, which can be applied to structure and grain size change of solids, sedimentation of isotope atoms, impurity control in semiconductors, etc. We mainly focus on the solid material processing and discuss the possibility of simulating the service environment of aerospace engines (Fig.1c) by this technique, since the state of solid sample in the high-temperature ultracentrifuge is very similar to the working condition of aerospace blades and turbine disks.

Mashimo[11–13] firstly proposed a self-consistent approach to the Lamm sedimentation equation for describing the diffusion induced by a strong gravitational field in a two-component condensed system. Sedimentation refers to the process of sinkage and accumulation of various substances in condensed matter, which can be caused by gravity, centrifugal force and electromagnetic force. Under the constant gravitational field (1 g) on the earth surface, the effect of sedimentation is very small and negligible. Multi-scale sedimentate behavior of particles will be influenced by gravity levels. The dirt and dust in the suspension are deposited at the bottom under the constant gravitational field. Furthermore, microparticles in liquid will be affected by the 10^4 g level gravitational field generated by the conventional ultracentrifuge machine. For

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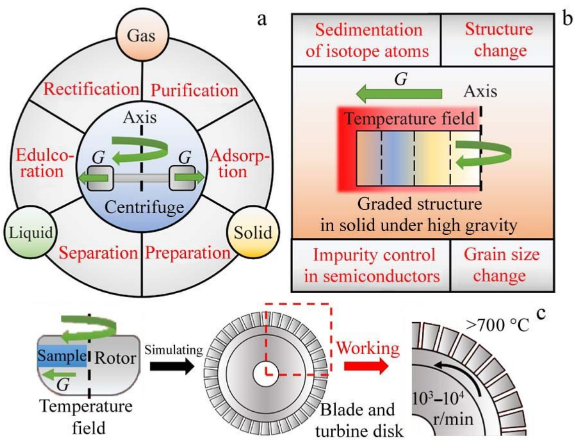


Fig.1 Schematics of three-phase mass transferring (a), solid material processing (b), and extreme service environment simulating of aerospace engine (c)

atoms in solids, the forces between tightly packed atoms are so strong that it possibly requires the mechanical energy of 10^6 g level gravitational field to overcome the chemical potential, which has been proved in recent experiments[14].

Just like electromagnetic field, gravity can be defined as a field-state variable which directly affects atoms through a kind of body force, whereas pressure and temperature are thermodynamic variables that affect atoms statistically. Under a strong gravitational field, the atoms will migrate in a specific direction due to the differences in mass and volume, which is the effect of sedimentation on composition. The migration of atoms results in changes in composition, affecting crystalline state, microstructure, dislocation motion, etc. By the sedimentation, materials can be controlled at the atomic-scale and new properties or new materials may be discovered. It is expected that the strong gravitational field will be used as a new method for atomic-scale materials processing to control the composition, impurities, nano-structure and interface structure of materials, and to concentrate isotopes[14]. Monoatomic solid Se was ultra-centrifuged at 190 °C under $(0.8-1)\times10^6$ g level, and the content of $^{82}Se/^{76}Se$ increases with gravity in different regions by >0.8%[15]. The lattice constants and binding energies of Se and Te continuously change along the direction of gravity in selenium-tellurium semiconductor at 260 °C under 10^6 g level[16].

This research outlines the tendency of sedimentation process in solids under a strong gravitational field in recent years, from the aspects of development of high-temperature ultracentrifuge and microstructure of ultra-centrifuged miscible systems and compounds material. Furthermore, the idea of applying the high gravity technique to simulate the service environment of aerospace engines was put forward.

1 Development of High-Temperature Ultracentrifuge

High-speed rotation of equipment is required to generate gravitational fields. In 1981, Ramshaw et al[17] developed the rotating packed bed (RPB) named as “HiGee” to enhance gas-liquid mass transfer. With the advantages of high efficiency, small size and low cost compared to conventional packed column, the rotor speed controls the contacting between gas and liquid in the cavity covered by the housing in Fig.2[18–20]. However, the gravitational field generated by the RPB is too small (10–1000 g level) to cause sedimentation process in solids (10^5–10^6 g level).

With regard to exploratory sedimentation research in smaller size solids, stable and strong gravitational field under high temperature for long term is necessary for overcoming the chemical potential between atoms. That is why the initial equipment tends to be developed on ultracentrifuges. Since Svedberg et al[21] developed the world's first turbo ultracen-trifuge in 1924, the maximum centrifugal force has reached 10^5–10^6 g level. However, the conventional ultracentrifuge is unable to operate under high temperature field for a long time. As for temperature, the high temperature makes atoms have higher energy and easier to transition, thus increasing the diffusion coefficient. Due to the requirements for the creep rupture strength of equipment materials, how to maintain a stable and high temperature field is an urgent problem to be solved. Therefore, we mainly focus on the gravitation and temperature value as well as their influencing factors.

By combining conventional ultracentrifuge with external heating, some types of interstitial diffusion were found in one of the initial experiments of sedimentation in solids performed on the ultracentrifuge (10^5 g, 300 °C and 4 d) developed by Atomic Energy Research Establishment, Harwell (UK)[22]. The rotor was heated by cold emission electron plasma in a low-pressure helium atmosphere, and the temperatures were measured by an infrared viewing thermometer. Since then, the prototype of high-temperature ultracentrifuge emerges.

From 1996 to 2001, Mashimo et al[23] of Kumamoto University developed an ultracentrifuge which could generate maximum acceleration field higher than 1.1×10^6 g at 100–300 °C for at least 85 h. It mainly consisted of a high-temperature air turbine motor for automobile and a specimen rotor driven by hot compressed air supplied by a screw compressor. Besides, two small specimen capsules were inserted on two symmetrical sides of the rotor (46 mm in outer diameter) made of titanium alloy (Ti-6Al-4V), whose strength

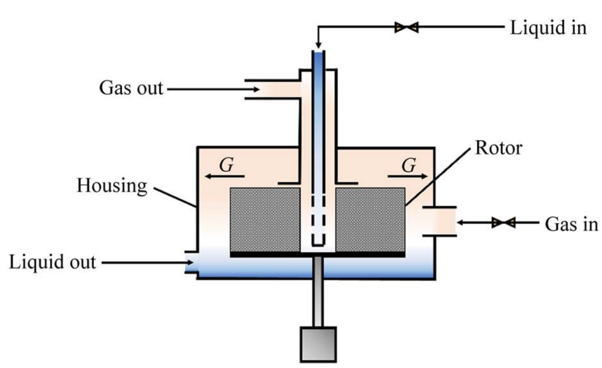


Fig.2 Schematic diagram of RPB[18]

limited the performance of the apparatus. It is really the first high-temperature ultracentrifuge used to study sedimentation in solids, which provides a platform for subsequent ultracentrifuge experiments.

From 2002 to 2010, the 2nd generation high-temperature ultracentrifuge, which could generate maximum acceleration field higher than 1.2×10^6 g over a wide temperature range up to >500 °C with high stability control, was updated by Mashimo et al[24] of Japan Atomic Energy Research Institute. Compared to the 1st generation, the big room-temperature air turbine motor with high control level was driven in the same way. Through the use of the electromagnetic air control valve and two turbine wheels, it is more precise to control the airflow and rotational speed. Besides, the heating of rotor changes from high-temperature air to Joule heating or radio-frequency heating in a vacuum, which extends the upper temperature limit to >500 °C while maintaining temperature stability. The most innovative part was the replaceable rotor whose outer diameter could be selected between 70 and 160 mm. The increase in outer diameter and material of rotor (Inconel 718) expand the gravitational field, energy range and sample scale. With the rotor as the most important part, the diagram of the high-temperature ultracentrifuge is shown in Fig.3.

In 2020, the cantilever high-temperature ultracentrifuge was developed for material processing and performance testing by Wei et al[25] of Zhejiang University, as shown in Fig. 4. Connected to the top drive spindle complex, the rotor system enters the experimental capsule through the lifting system and is rotated by the centrifugal main engine. So far, the tested max gravity and temperature have reached 1.0×10^5 g and 1200 °C, respectively. The rotor diameter is 50–1800 mm, more than 10 times of the previous one. Compared to the former high-temperature ultracentrifuges, though the max gravity is one order of magnitude lower, the max temperature covers the application scope of some superalloys, providing equipment support for simulating aerospace engine service environment. We plan to carry out the high gravity experiment of superalloy on this equipment in the future.

The differences among the 1st generation high-temperature ultracentrifuge of Kumamoto University, the 2nd generation of JAERI and the high-temperature ultracentrifuge of Zhejiang University are summarized in Fig. 5[23–25]. Through comparative analysis, it can be found that the overall

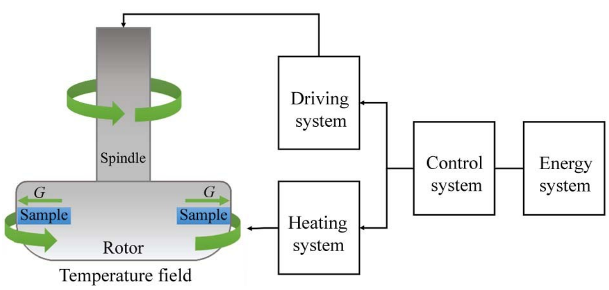


Fig.3 Diagram of the high-temperature ultracentrifuge

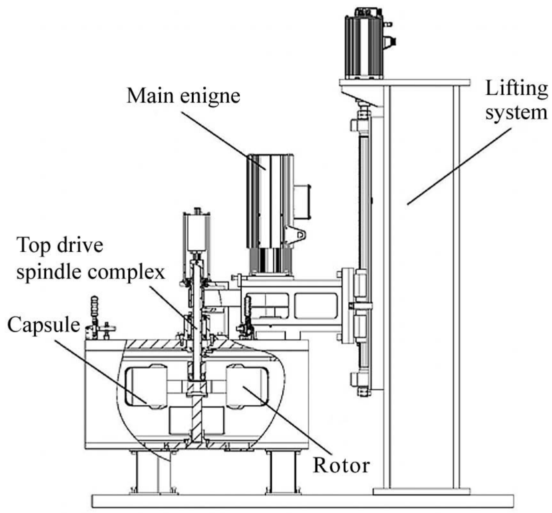
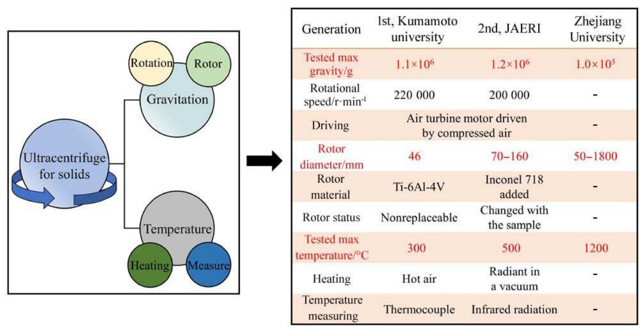


Fig.4 Cantilever type centrifugal hyper gravity experimental device for material preparation and performance test[25]



Generation	1st, Kumamoto university	2nd, JAERI	Zhejiang University
Tested max gravity/g	1.1×10^6	1.2×10^6	1.0×10^5
Rotational speed/r·min^{-1}	220 000	200 000	-
Driving	Air turbine motor driven by compressed air		-
Rotor diameter/mm	46	70–160	50–1800
Rotor material	Ti-6Al-4V	Inconel 718 added	-
Rotor status	Nonreplaceable	Changed with the sample	-
Tested max temperature/°C	300	500	1200
Heating	Hot air	Radiant in a vacuum	-
Temperature measuring	Thermocouple	Infrared radiation	-

Fig.5 Differences between three generations of high-temperature ultracentrifuges[23–25]

performance of the equipment is improved, mainly reflected in the rise in the maximum value and the decline in the fluctuation of various parameters. For gravitational experiments requiring high-speed rotation for a long time, maintaining the stability of the strong gravitational field and temperature field is conducive to eliminating errors caused by fluctuations. The upgrading of the rotor expands the coverage of materials.

In a word, the development of high-temperature ultracentrifuges is mainly reflected in the rotation rate, rotor radius and temperature. The material of the equipment has been gradually replaced by superalloys with high creep strength to ensure the strength under strong gravitational field and high temperature field. The upgrade of other hardware and software also promotes the improvement of parameters. A prototype rotor with two grooves was developed for the multistage centrifugal isotope separation in solid state[26]. Due to the complex system, great difficulty and long process in research and development of high-temperature ultracentrifuge, only specific materials, miscible systems and compounds have been tested. Large volume, poor flexibility, complex system and other shortcomings still make the existing high-temperature ultracentrifuges huge and not intelligent. Except improving the range and stability of the strong gravitational field and temperature field, the integration of equipment should also be enhanced.

2 Research on High Gravity of Solid Materials

2.1 Miscible systems

The initial experiments of sedimentation in solids are Au isotopes in metals with a low melting temperature (K, In and Pb) under a strong gravitational field[22,27–28]. The strong gravitational field was used to improve the theoretical model of rapid diffusion of noble metals in other metals where interstitial solutes act. Due to the low concentrations of Au isotope, at an impurity level, the concentration varies little, only supporting hypotheses based on some types of interstitial diffusion without further explanation.

From then on more experiments about miscible systems (mainly binary alloys) have been performed to explore the characteristics caused by high gravity and sedimentation mechanisms, as summarized in Fig.6[16,29–39]. The strong gravitational field, in which the experiment is conducted, is usually within a range, so the maximum gravity value is taken for statistics. It can be concluded that the max gravity is $(0.12-1.02)\times10^6$ g, and the temperature field does not exceed 400 °C, because the melting points of the mainly studied binary alloys are relatively low. Nearly half of the experiments were conducted for Bi-Sb alloys which are the best n-type thermoelectric materials for refrigeration at low temperatures[40–41].

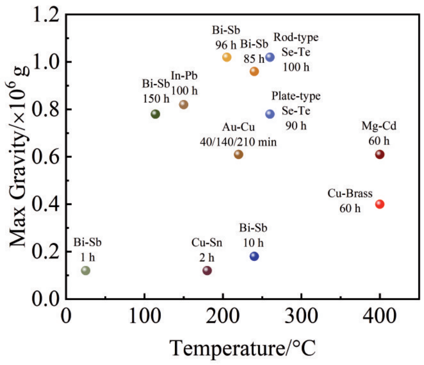


Fig.6 Ultracentrifuge experiments for sedimentation in miscible systems[16,29–39]

In 1997 and 2001, Bi-Sb alloy was tested at 220–240 °C for 85 h under the gravitational field of $(0.79-0.96)\times10^6$ g by Mashimo et al[29–30]. The lattice constants of a_0 and c_0 are dropped constantly as the gravitational field decreases, as shown in Fig.7a, while the composition change of Bi and Sb is consistent with that calculated by the lattice constants according to Vegard's law, as shown in Fig. 7c. The composition and lattice constant continuously change at the atomic scale, resulting in graded structure, which provides the evidence of sedimentation of substitutional solute atoms in solids. The simulation program of the sedimentation process is based on a self-consistent theory, indicating the difference between sedimentation and vacancy mechanism for sedimentation with very large diffusion coefficient[42]. Mashimo et al[31] used In-Pb alloy and got an atomic-scale graded structure similar to Bi-Sb alloy, the same as the Mg-Cd alloy[33]. The crystal growth was observed in the strongest gravitational field along the direction in Bi-Sb alloy, as shown in Fig.7b[30]. In 2004, Huang et al[32] conducted a more thorough investigation on the Bi-Sb alloy with an emphasis on grain size. Three experiments were performed on Bi-Sb alloy, and the grain refinement and crystal growth were affected by low and high gravity regions. Strong correlations were found between the growth of crystals in the solid state and sedimentation of atoms. It is proposed that following grain

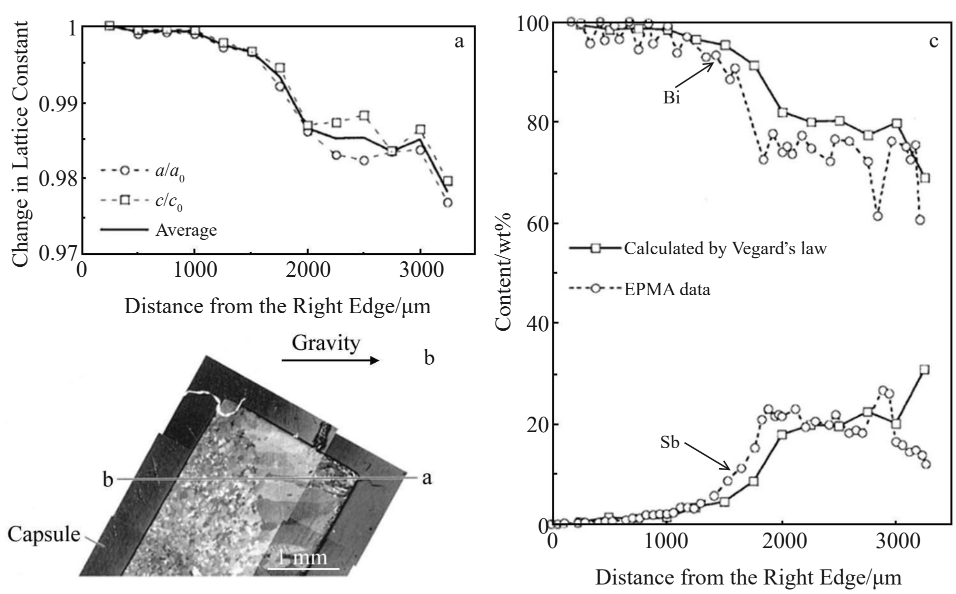


Fig.7 Change in the lattice constants of *a* and *c* axes (a); polarization of crystal growth area (b); composition profile of Bi and Sb obtained by EPMA data and Vegard's law (c)[27]

refining, the strongest gravitational field serves as the starting point for crystal growth as well as sedimentation of atoms.

Despite the results suggesting that sedimentation plays a role, there is still a lack of basic understanding on how it works. In order to focus on the interaction between the direction of strong gravity and the atoms at the metal interface, the direction of applied strong gravity and the type of miscible systems have been broadened.

In 2012 and 2015, Ogata et al[36–37] made a major discovery in sedimentation mechanism by centrifuging Cu-Brass diffusion couples at 0.4×10^6 g and 400 °C for 60 h with interfaces normal to the direction of gravity, as shown in Fig. 8. According to the EPMA concentration distributions, it is found that after annealing under different gravity fields, the composition gradient at the interface between copper and brass becomes broader: normal sample I>1 g sample>normal sample II>initial sample, which means that the diffusion in normal sample I is enhanced by strong gravitational field while that in normal sample II is decreased. Combining diffusion and atomic migration, the sedimentation mechanism is assumed as follows: the denser Cu atoms diffuse in the direction of gravity and the less dense Zn atoms diffuse in the opposite direction under sedimentation of atoms. Also, the fraction of three phases in Cu-Sn alloy, Cu_3Sn, Cu_6Sn_5 and pure Sn, changes with the sedimentation process[39]. It is indicated that the less dense Sn atoms migrate to the center of the axis rotation in the opposite direction of the centrifugal force, which strongly supports the sedimentation mechanism of atomic migration.

Moreover, vacancies are formed throughout the parallel-mode sample in Cu-brass diffusion couples and migrate to the lower gravity region by sedimentation[37]. As atoms are moved by body forces in the strong gravitational field and high temperature field, dislocations and a large number of vacancies might be continually produced. These voids have no mass, and their sedimentations occur at the area of lower gravity. The implication is that voids created in the higher gravity region have transferred to the lower gravity region through sedimentation, thus increasing the values of the diffusion coefficients. Inner cracks are also found in Bi-Sb alloy under 1.20×10^5 g at room temperature for 1 h, which can be caused by the accumulation of voids[38]. The high gravity is regarded as tension in Cu-Sn couple, which inhibits the rate of diffusion and formation of two intermetallic layers due to more gaps and less Kirkendall pores[43].

As the mostly studied miscible systems, binary alloys have simpler microstructures than multielement alloys, making atomic migration and composition changes more visible under a strong gravitational field. By studying the overall states and interface of miscible systems, it can be found that the material exhibits an atomic-scale graded structure due to the strengthening of the strong gravitational field in steps from the axis to the outside, including composition change, grain growth, refinement, voids accumulation, etc. The summarization of such phenomena indicates the sedimentation mechanism of atomic migration: the denser atom migrates in the direction of gravity, and the less dense atom migrates in the opposite direction of gravity. However, whether this mechanism is universally applicable to other multiple systems still needs to be tested.

2.2 Compounds

Changes in the stoichiometry and structure of the material

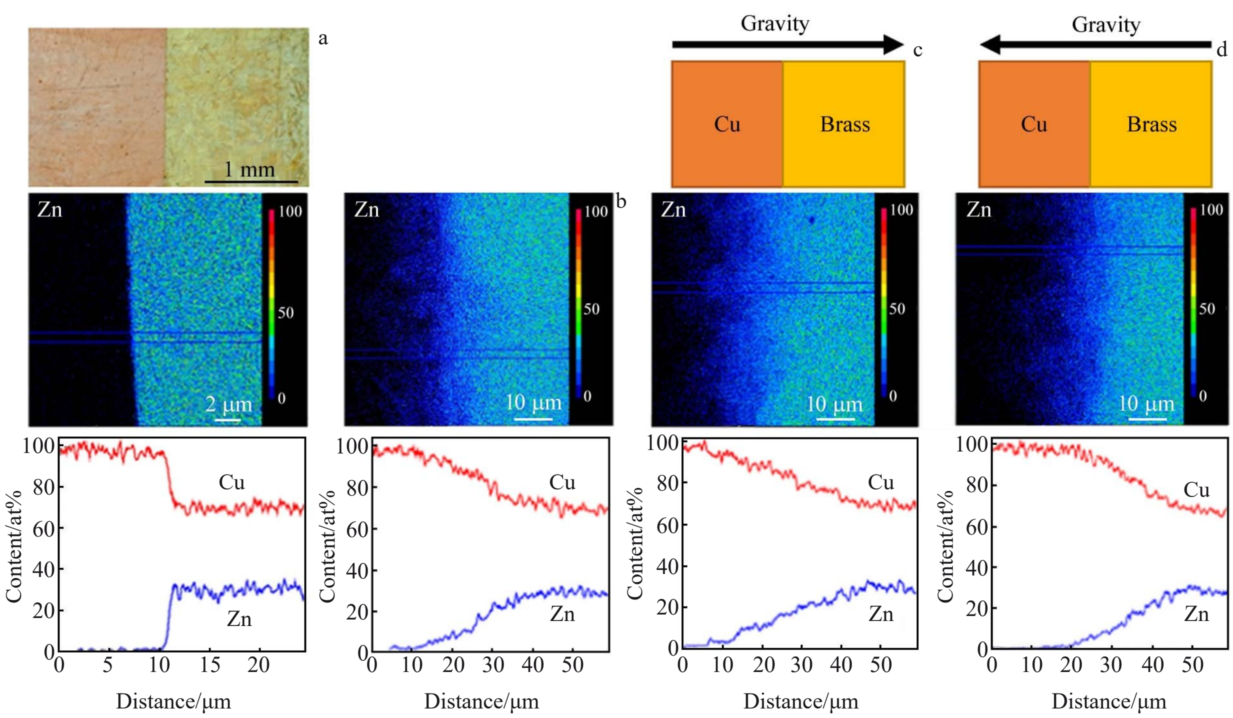


Fig.8 EPMA concentration distributions of initial sample (a), 1 g sample (b), normal sample I (c) and normal sample II (d)[36–37]

are anticipated in the presence of the strong gravitational fields produced by the ultracentrifuge, which may be beneficial to new phases generation with distinct properties[44]. The effects of strong gravitational field on compounds are summarized in Fig. 9[44–49]. Using the same high-temperature ultracentrifuges, the range of max gravity and temperature is similar to that of the miscible systems, because the selection of parameters should match with equipment. There are $La_{1-x}Sr_xMnO_3$, TiO_2 and V_2O_5 when the parameters are 0.4×10^6 g, 400 °C and 24 h. The selected compounds are mainly intermetallic compounds and metal oxides with super-conducting property, magnetoresistance effect and other characteristics caused by unique structure. Under the strong gravitational field, the structure of compounds may be affected, resulting in new structures, properties and substances.

The atomic-graded structure and former sedimentation mechanism are also appropriate to compounds unquestionably. There is a visible four-layers structure in a thin-plate sample (0.7 mm in thickness) of Bi_3Pb_7

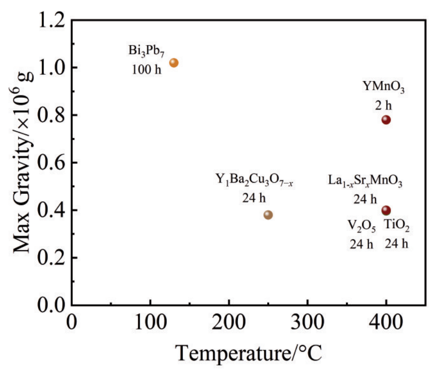


Fig.9 Ultracentrifuge experiments for sedimentation in compounds[44–49]

ultracentrifuged at 1.02×10^6 g and 130 °C for 100 h[45]. The composition-graded structure is observed at the 2nd and 3rd layers, where Pb content increases along gravity direction. Another superconducting material $Y_1Ba_2Cu_3O_{7-x}$ (Y123) was also divided into lower and higher gravity layers after ultra-centrifuging at 0.38×10^6 g and 250 °C for 24 h[44]. The composition is uniform in the initial sample, but atomic-scale composition-graded structures appear after the strong gravity treatment. Both Ba and Cu contents decrease along the direction of gravity, while Y content increases along the direction of gravity. Though the atomic mass of Y is smaller than that of Ba and Cu, the atomic volume of Y is much smaller than that of Ba and Cu in YBCO crystals, which proves once again that the sedimentation mechanism under the strong gravitational field is related to the density of atoms, not just the mass or volume.

Moreover, structure changes and decomposition occur, which causes the growth of large, green and transparent Y_2BaCuO_5 crystals in the higher gravity layer and improves the properties of $Y_1Ba_2Cu_3O_{7-x}$[44]. In addition, Khandaker et al[47] put an orthorhombic V_2O_5 polycrystal phase to strong gravitational field at 0.397×10^6 g and 400 °C for 24 h, and a graded structure was produced by the gravity-induced com-positional change, as shown in Fig.10. The XRD and Raman spectra indicate that monoclinic VO_2 phase is introduced into the low gravity region and converted into monoclinic V_2O_3 phase near the high gravity field. The strong gravitational field causes a type of graded vanadium oxide structure that benefits from the positive traits of its constituent V_2O_5, VO_2 and V_2O_3.

Mashimo et al[48] reported the preparation of single-crystal rutile (TiO_2) with a unique structure prepared by ultracen-trifugation at $(0.39-0.40)\times10^6$ g and 400 °C for 24 h, which was summarized as the uniaxially distorted crystalline state, as shown in Fig. 11. Under pressure, the lattice shrinks

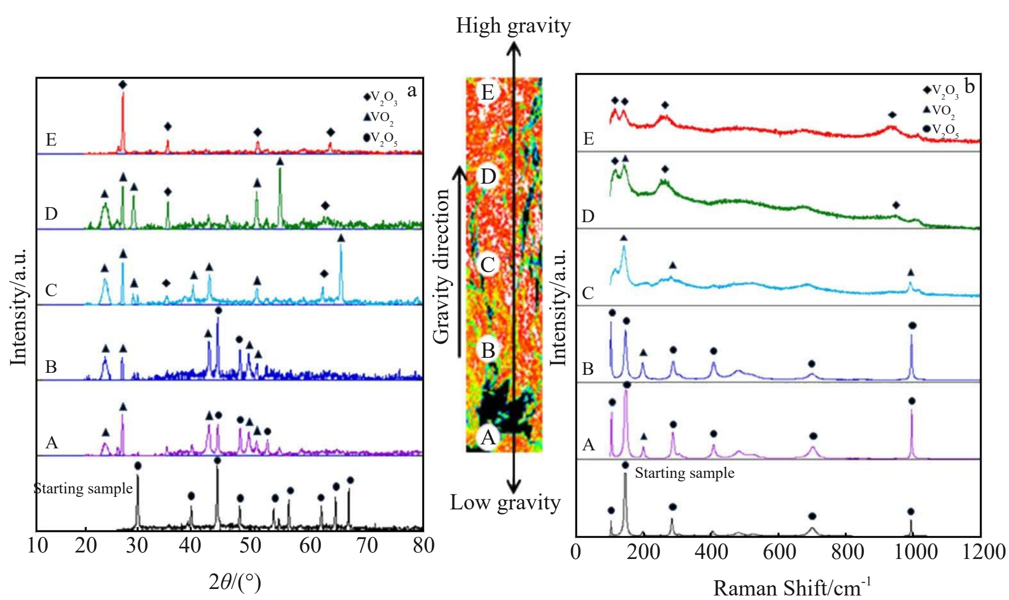


Fig.10 XRD patterns of a micro area (0.1 mm) on a polished surface of the starting sample and gravity sample (a) and corresponding Raman spectra (b)[47]

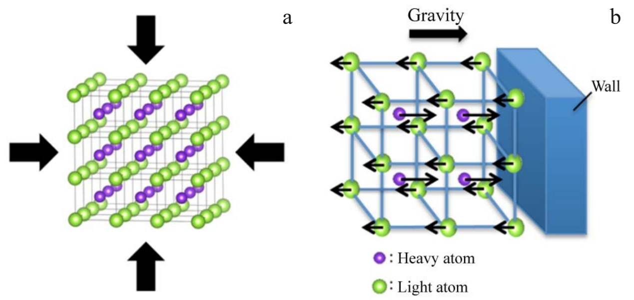


Fig.11 Schematic diagrams of crystal structures of compounds under high pressure (a) and strong gravitational field (b) where the crystal position is restrained by a wall[48]

isotropically according to the equation of the state of matter. However, under a strong gravitational field, different body forces act on the respective atoms owing to their different atomic masses and volumes, i. e., heavy atoms are forced in the gravitational direction and light atoms are forced in the opposite direction, showing a uniaxially distorted crystalline state. The similar phenomena have also been found in hexagonal $YMnO_3$ single crystal at 0.78×10^6 g and 400 °C for 2 h, demonstrating that strong gravitational field can yield unique distorted structure and magnetic properties, which cannot be obtained by annealing treatments and the high-pressure processing of $YMnO_3$[49].

Through the above research on miscible systems, a conclusion of the sedimentation mechanism under the strong gravitational field, i. e., gravity-induced atomic migration, is proposed. Under the strong gravitational field, the denser atoms migrate in the direction of gravity, and the less dense atoms migrate in the opposite direction of gravity, since the gravity is a kind of body force. It can be seen that the conclusion summarized for the miscible systems is also applicable to the compounds.

While being applied to the research of compounds, this conclusion has absorbed the achievements of compounds and is constantly improved. By associating the structural characteristics of compounds with the gravity-induced atomic migration, it can be concluded that the migration of atoms leads to changes in atomic bond distance, angle and position, which makes the crystal present a unique uniaxially distorted crystalline state and thus affects the properties of compounds. The research on sedimentation process in solids under high gravity is summarized in Fig. 12. Due to the desire to create new compounds, the possibility of applying strong gravity technique to create new compounds is investigated more, and it has indeed succeeded. This will be the first step for the application of high gravity technique to change the characteristics of solid materials.

2.3 Superalloys

The emergence of high-temperature ultracentrifuge has made it possible to simulate the high-temperature and high-speed service environment of workpieces. Nickel-based superalloys are frequently used as turbine blade and disk for

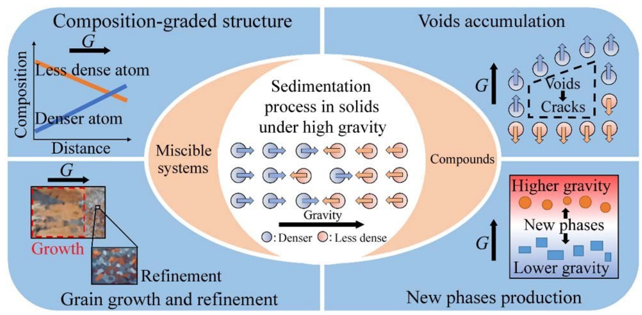


Fig.12 Summarized researches on sedimentation process in solids under high gravity

industrial gas turbines and aerospace engines because of their excellent high-temperature strength, creep and fatigue resistance and oxidation and hot corrosion resistance[50–51]. As for aerospace engines, parts of engine rotates at high speed while being subjected to high temperature, which tests the high-temperature mechanical properties of materials. Currently, creep resistance is mainly used to evaluate the life of superalloys in service environments of high-temperature and high-speed, such as engines. However, the sample is subjected to tensile stress constant in both value and direction in the creep and rapture testing machine, while the actual centrifugal tensile stress direction continuously rotates and changes.

We mainly focus on the high-temperature mechanical properties of tensile property, creep resistance and fatigue property as well as their testing methods, as summarized in Fig. 13. The existing testing methods for high-temperature mechanical properties, including tensile stress, compressive stress, cyclic stress, etc, mainly apply temperature field and stress field to superalloy materials. Because the stress is a kind of internal surface force on the unit sectional area of the applied material, the material will be under the coupling action of "surface force+temperature". However, when the engine rotates at high speed, it is subjected to not only the internal stress but also the high gravity with direction changing at any time. High gravity is a kind of body force on

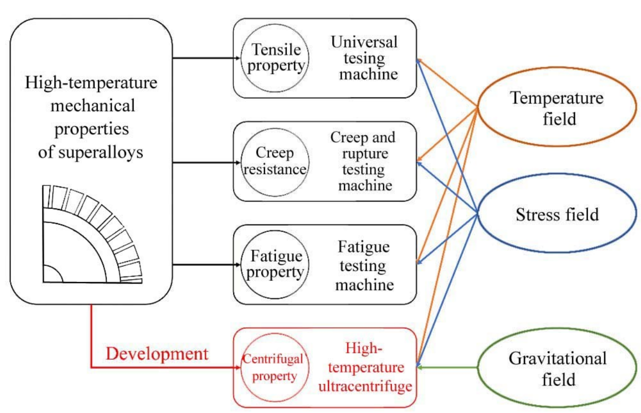


Fig.13 High-temperature mechanical properties and testing methods of superalloys

the volume elements of the applied material, so the material will be under the coupling action of “body force+surface force+temperature”, which is the most similar to the aerospace engine service environment. By characterizing and analyzing the nickel-based superalloy blades of compressor and gas turbine operated more than 10 000 h, unique phenomena occur, such as segregation at the zigzag-like grain boundary and nonhomogeneous microstructural degradation, which might be caused by the long-period centrifugal force[52–53]. Using the high-temperature ultracentrifuge, Liu et al[54] studied the failure mechanism of single crystal superalloy with thin film cooling holes under high-speed rotation and high-temperature coupling conditions. With the support of high-temperature ultracentrifuge, more realistic high-temperature mechanical properties of material, i.e., centrifugal property, will be tested and evaluated.

Composition and microstructure have an impact on the properties of material. It is essential to pay attention to the change of material composition and microstructure in the application and to assess its impact on properties. Take the widely studied and applied nickel-based superalloy, Inconel 718, as an example, its application temperature range is generally below 650 °C. The aging process of Inconel 718 shows that above 650 °C, with the increase in aging time, the main strengthening γ'' phase will change to δ phase, making the mechanical properties at high temperature drop rapidly[55–56]. This is the phase transition mechanism of Inconel 718 under constant gravity. However, we have already conducted high gravity treatment on Inconel 718 and found that high gravity has an inhibitory effect on the precipitation of δ phase. The high gravity alters atomic diffusion, especially for Nb element which is the main forming elements of precipitates, thereby altering element segregation and affecting the conversion of the main strengthening γ'' phase to the harmful δ phase. In addition, the multi-field coupling effects on composition and microstructure of Inconel 718 were studied, including stress field, magnetic field, electric field, etc[57–62].

On the one hand, the strong gravitational field can change the composition and structure of materials; on the other hand, the study of the composition and structure of the ultracentrifuged sample is helpful to understand the distribution of the gravitational field and the stress field caused by it. At a constant speed, high gravity increases with the increase in radius, and the direction changes constantly. The resulting stress field is more elusive. At present, the high gravity experiment is still in the stage of setting process parameters to observe the changes of composition and microstructure. It is expected to summarize the action law of the force field and to apply it through continuous tests.

3 Summary and Perspective

In this study, the relevant progresses about sedimentation in solids under a strong gravitational field are described, mainly for equipment, miscible systems and compounds. The self-consistent approach to the Lamm sedimentation equation proposed by Mashimo provides a theoretical basis for the experiments, and the high-temperature ultracentrifuge is also indispensable. Since Mashimo developed the 1st and updated the 2nd high-temperature ultracentrifuge, almost 90% ultracentrifugation experiments in solids have been performed on them. So far, experiments of ultra-centrifugal sedimentation in solids are mainly carried out in miscible systems and compounds, which is in subservience to explore the mechanism of sedimentation. The conclusions can be expressed as follows.

1) The two most important parameters provided by high-temperature ultracentrifuges, i. e., rotational speed (strong gravitational field) and high temperature field, restrict experiments of ultra-centrifugal sedimentation in solids, and the upper limit of temperature field is not high enough.

2) The phenomena of atomic-scale graded structure (composition and lattice constant), grain growth and refinement, voids accumulation, new phases production and uniaxially distorted crystalline states in miscible systems and compounds can all be attributed to sedimentation of atoms in solid under high gravity.

3) High gravity directly affects atoms through a body force: the denser atoms diffuse in the direction of gravity and the less dense atoms diffuse in the opposite direction. The diffusion coefficient increases due to the migration of atoms, which is called sedimentation (gravity-induced diffusion).

As an emerging material processing technique, strong gravity in solids has been applied in grain refinement for binary alloys, structure change in compounds, impurity control in semiconductors, sedimentation of isotope atoms, etc, showing great potential. The application of high gravity can also be extended to simulate extreme environment. High rotation speed and high temperature field generated by the high-temperature ultracentrifuge can be used to simulate the service environment of aerospace engines, which will be instructive for practical engineering applications. Extensive and in-depth research on sedimentation process in solids under a strong gravitational field has seldom been carried out and a large number of scientific questions remain to be explored.

1) The present sedimentation theory is self-consistent and phenomenological, which needs to be improved and deepened. There is a lack of research and modeling of microscopic mechanisms, such as diffusion rates, diffusion coefficients and mechanisms of formation of graded structures.

2) The characterization of the sample is only on a two-dimensional plane, and the resulting changes in composition are two-dimensional, which cannot fully demonstrate the impact of a three-dimensional gravitational field on the sample.

3) When studying the same material, it is rare to use variables-controlling method to study the effect of different gravitational accelerations and temperatures on sedimentation in solids, and the gravitational field and temperature cannot be controlled to obtain the desired properties.

4) The application of high gravity technique in multi-

component superalloys is still blank. The three-in-one system of material processing, performance testing and environment simulating for superalloys in the high-temperature ultracentrifuge is gradually built.

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超重力下的沉降过程与固态相变

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摘　要：介绍了超离心状态下固体中沉降过程的最新进展以及未来应用前景，主要涉及高温超速离心设备、互溶体系以及化合物材料中的超重力固态相变。近90%的固态超速离心实验是在世界第一台（熊本大学）与第二台（日本原子能研究所）高温超速离心机上，以500 ℃以下的温度与10^6 g的最大离心加速度条件运行。强大的超重力场和温度场诱导材料中出现原子级梯度结构、晶粒生长与细化以及空位聚集等现象，并产生了新的物相结构与特性。浙江大学建造了一种实验温度高达1200 ℃的新型悬臂式高温超速离心机，为模拟高温合金在航空发动机运行环境中的合金成分、微观结构和性能演变提供了设备基础。

关键词：超重力；沉降；扩散；高温合金

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