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The role of zirconium in the formation of structure and properties of titanium alloys during superplastic deformation

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Abstract. This study investigates the effect of zirconium on the superplastic properties of titanium alloys at various temperatures and strain rates. It has been established that zirconium significantly influences the strain rate sensitivity coefficient (m), mechanical stability, and plasticity. At elevated temperatures, zirconium-containing alloys exhibit a stable m-value within a specific strain rate range, followed by a sharp decline. In contrast, zirconium-free alloys show a gradual decrease in m as the strain rate increases. The optimal temperature-strain rate conditions for superplastic deformation depend on zirconium content. Alloys with lower zirconium concentrations demonstrate high plasticity at moderate temperatures and intermediate strain rates, whereas alloys with higher zirconium content require lower strain rates to achieve uniform deformation. Beyond a certain threshold, an increase in zirconium content results in reduced plasticity and strain localization. Additionally, zirconium increases flow stress, while higher temperatures contribute to its reduction; however, this is accompanied by grain coarsening, which negatively affects mechanical properties. Microstructural analysis using scanning electron microscopy revealed that after superplastic deformation, all investigated alloys develop a fine-grained structure consisting of equiaxed α - and β -grains. The average grain size increases compared to the initial state, indicating dynamic recovery and recrystallization processes. The results confirm the feasibility of using these titanium alloys in superplastic forming technologies. The identified correlations provide a basis for optimizing thermomechanical processing parameters to achieve a balance between high plasticity and mechanical stability, which is crucial for industrial applications.

Keywords: ultrafine-grained, severe plastic deformation, structure, nanoscale, superplasticity, zirconium.

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1. Introduction

Severe plastic deformation (SPD) is one of the most effective methods for forming an ultrafine-grained (UFG) structure in metals and alloys. This approach significantly improves the mechanical properties of materials by reducing the average grain size and increasing the density of crystal lattice defects. As a result, SPD-processed materials exhibit enhanced strength, fatigue limit, and superplasticity, making them promising for applications in various industries [1-4].

In recent years, various SPD techniques have been developed to refine the structure of titanium alloys efficiently. The most common methods include equal-channel angular pressing (ECAP), high-pressure torsion, high-cycle impact treatment, and multistep forging. Studies have shown that SPD by torsion can produce an ultrafine-grained structure with an average grain size of approximately 80 nm [5-7]. In turn, multistep forging performed within the temperature range of 800–400 °C facilitates the formation of equiaxed grains with an average size of about 200 nm [8-13]. The ECAP method, applied to bulk billets, enables the achievement of a grain size of approximately 260 nm after eight processing cycles [14-17].

However, despite the significant increase in strength, the formation of a UFG structure is often accompanied by a reduction in impact toughness and fracture resistance, which may limit the application of such materials in structural components. To address this issue, specialized UFG structures are being actively developed to achieve an optimal balance between strength and fracture toughness [18-21]. One promising approach involves creating microstructural composites consisting of «hard» and «soft» phases. In two-phase titanium alloys, the α -phase enhances plasticity due to the presence of multiple slip systems [22, 23].

Controlling the shape, volume ratio, and spatial distribution of these phases plays a key role in regulating the material's mechanical properties. The introduction of alloying elements enables targeted modifications of the phase composition and microstructure, enhancing the material's performance characteristics. One promising direction for improv-

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UFG titanium alloys are of significant interest for applications under superplastic deformation conditions. Twophase titanium alloys are at the center of modern research aimed at developing technological processes that utilize their superplastic properties. It is important to note that optimizing the temperature range for superplasticity (150-400°C) is crucial for ensuring the high manufacturability of such materials [25]. The application of superplasticity at lower temperatures significantly reduces energy consumption while maintaining high mechanical properties, making this process particularly attractive for industrial manufacturing. For example, low-temperature superplasticity improves the strength characteristics and enhances the quality of thin-walled components formed in complex geometries [26, 27].

The main mechanisms of superplasticity include grain boundary sliding, intragranular dislocation slip, diffusion creep, and dynamic recrystallization. A key characteristic of superplastic flow is grain size: the finer the grain, the higher the material's strain rate sensitivity, the lower its yield strength, and the greater its deformation capacity. This is why an ultrafine-grained structure significantly enhances superplasticity efficiency [28-46].

Additionally, studies indicate that metallic glasses can exhibit superplastic behavior at relatively high strain rates. This effect opens new prospects for developing materials with high technological plasticity, capable of withstanding significant loads without failure [47].

In prototype studies, the mechanical behavior of materials under superplastic conditions is examined with a focus on uniaxial tension, compression, and torsion under applied pressure, as well as punching and compression in specially designed dies. Based on deformation diagrams and characteristic curves, such as the relationship between true yield stress, strain rate sensitivity, maximum plasticity, and other factors as functions of strain rate, it is assumed that superplastic behavior under certain temperature-strain rate regimes is reasonable [28-47].

Various types of tests are conducted to study the mechanical behavior of materials under superplasticity, including uniaxial tension, compression, torsion under pressure, and material extrusion in dies. Essential aspects of the research include constructing deformation diagrams and characteristic curves, which help characterize the adaptive properties of the material and its superplastic behavior under different temperature and strain rate conditions. In particular, the dependencies of true flow stress $\sigma_y(a)$, strain rate sensitivity coefficient

$$m(a)$$
, and maximum elongation at fracture $\delta(a) = \frac{l}{l_0} - 1$ on

the strain rate can be described by the following equations:

$$\alpha \coloneqq \dot{\varepsilon} = l/l = V/l, \tag{1}$$

where, $\varepsilon = \ln l / l_o$; *V* – the crosshead speed of the testing machine. These curves are typically plotted in (semi-) logarithmic coordinates:

$$\lg \sigma - \lg a, m - \lg a, \delta - \lg a , \tag{2}$$

Based on their qualitative appearance, these curves can determine whether a material is in a superplastic state or exhibits conventional deformation behavior. Superplastic materials are characterized by specific deformation curves, where an initial sharp increase in stress is followed by a plateau with constant stress (without strain hardening), and in some cases, even a decrease in stress.

Alloying elements such as zirconium (Zr), chromium (Cr), and manganese (Mn) play a crucial role in achieving superplasticity. They influence plastic deformation mechanisms, enhancing structural stability and increasing plasticity. Studies have shown that aluminum-magnesium alloys with zirconium additions exhibit significant improvements in superplasticity due to the stabilization of the microstructure by dispersed Al₃Zr particles. These particles prevent grain growth at high temperatures, ensuring stable superplasticity up to $525^{\circ}C$ [48]. For example, a modified Al-Mg-Zr alloy at $500^{\circ}C$ demonstrates a maximum elongation of 1013% at a strain rate of $5 \cdot 10^{-2}$ s⁻¹, making it a promising material for applications requiring high plasticity [48].

The introduction of zirconium into titanium and aluminum alloys significantly lowers the temperature for superplastic deformation and increases the strain rate at which high plasticity is maintained. This improves the material's processability, reduces energy consumption during processing, and enhances the efficiency of forming processes.

Studying the dependence of the strain rate sensitivity coefficient on strain rate and temperature is an important aspect of research, as it helps determine optimal temperature-strain rate conditions that maximize superplasticity. This, in turn, plays a key role in improving manufacturing efficiency and expanding the use of materials with enhanced properties in industrial applications.

2. Materials and methods

The study examined alloys with varying zirconium content: 0Zr, 0.5Zr, 1Zr, and 1.5Zr. Samples with the geometry shown in Figure 1 were cut from 1 mm-thick titanium sheets along the rolling direction.



Figure 1. Sample geometry for superplasticity testing

The samples were tested using a ZWICK 250 universal testing machine.

To determine the deformation conditions (strain rate and temperature) for superplasticity testing, step strain rate tests were conducted at temperatures of 700, 725, and 750°C. The strain rate varied within the range of $5 \cdot 10^{-5}$ s⁻¹ to $2 \cdot 10^{-2}$ s⁻¹, with a step increment of $2.5 \cdot 10^{-5}$ s⁻¹ every 2% strain.

Based on the results of the step strain rate tests, the strain rate sensitivity coefficient (m) was determined.

$$m = \partial L n \sigma / \partial L n \varepsilon , \qquad (3)$$

The microstructure of the obtained samples was analyzed using a TESCAN VEGA 3 scanning electron microscope.

3. Results and discussion

The study of the superplasticity of the alloys was conducted at constant strain rates, where both strain rate and temperature were set based on preliminary step strain rate tests. These tests allowed for determining the limit values of the strain rate sensitivity coefficient (m), which plays a key role in evaluating a material's superplasticity. The m coefficient characterizes the degree of dependence of flow stress on strain rate and, accordingly, indicates the dominant plastic deformation mechanism.

Figure 2 presents the dependence of the strain rate sensitivity coefficient of the studied alloys on strain rate at 700, 725, and 750°C.



Figure 2. Dependence of the strain rate sensitivity coefficient of the alloys on strain rate at different temperatures (°C): (a) - 700; (b) - 725; (c) - 750

At 700°C, alloys containing zirconium (0.5Zr, 1Zr, 1.5Zr) show a similar dependence of the m coefficient on strain rate. In the strain rate range up to $2 \cdot 10^{-3}$ s⁻¹, the m coefficient

remains nearly constant, indicating stability of the superplastic state under these conditions. This suggests that within this strain rate range, the dominant plastic deformation mechanism is grain boundary sliding (GBS), which is active at high grain boundary diffusion mobility.

However, when the strain rate exceeds $2 \cdot 10^{-3} \text{ s}^{-1}$, a sharp decrease in the m coefficient is observed. This indicates a transition from a superplastic regime to a conventional plastic deformation regime, associated with the activation of dislocation slip and subsequent material hardening. It is likely that at high strain rates, grain boundary sliding can no longer accommodate plastic deformation, leading to a decrease in m.

At the same time, the zirconium-free alloy exhibits a monotonic decrease in the m coefficient with increasing strain rate. This may be due to the lower superplasticity capability of this alloy, possibly related to limited grain boundary mobility and a more pronounced dislocation-based deformation mechanism.

At 700°C, alloys containing zirconium (0.5Zr, 1Zr, 1.5Zr) exhibit similar values of the strain rate sensitivity coefficient m as a function of strain rate. In the strain rate range below $2 \cdot 10^{-3}$ s⁻¹, the coefficient m remains nearly constant, indicating the stability of the superplastic state under these conditions. This behavior is associated with the dominant grain boundary sliding (GBS) mechanism, which, at moderate strain rates, ensures uniform redistribution of plastic deformation without significant material hardening. However, as the strain rate increases beyond $2 \cdot 10^{-3}$ s⁻¹, the m value begins to decrease significantly, signaling a gradual transition of the material from a superplastic state to conventional deformation behavior, characteristic of dislocation slips mechanisms. This phenomenon occurs because, at high strain rates, grain boundary sliding does not have sufficient time to accommodate localized plastic deformation, leading to the activation of hardening processes.

At 725°C, all studied alloys exhibit high m values exceeding the required superplasticity threshold (0.3). The dependencies of m on strain rate are similar to those observed at 700°C, but with a wider strain rate range ensuring stable superplastic behavior. For alloys containing 0.5-1.5% Zr, the maximum m values are reached at strain rates around $2 \cdot 10^{-3}$ s⁻¹, which corresponds to an optimal balance between grain boundary sliding and dislocation processes.

The highest efficiency of zirconium in promoting superplastic deformation is observed at 750°C. In this temperature range, all studied alloys exhibit similar m dependencies on strain rate. The greatest increase in m is noted at a strain rate of $2 \cdot 10^{-3}$ s⁻¹, where the maximum value reaches approximately 0.35.

The addition of zirconium significantly influences the mechanical properties of titanium alloys by stabilizing the fine-grained structure.

Superplasticity studies enabled the determination of elongation values for different alloys under various temperaturestrain rate conditions, where the strain rate sensitivity coefficient m approaches its maximum values.

Figure 3 presents the tensile curves for the alloys at different temperatures and strain rates. All investigated alloys demonstrate superplastic behavior, with elongation exceeding 200% under the selected deformation conditions, confirming a significant potential for superplastic deformation.



Figure 3. Tensile curves of alloys: a) 0Zr, b) 0.5Zr, c) 1Zr, and d) 1.5Zr at a temperature of 725 °C

However, as the specific zirconium content in the alloy exceeds 0.5%, elongation increases, but deformation becomes less uniform, and the flow stress rises. Alloys with a lower zirconium content exhibit a weaker dependence on strain rate and temperature, indicating better superplasticity in these compositions.

In contrast, when the zirconium content increases to 1% or more, a sharp decrease in elongation is observed with increasing strain rate, indicating a deterioration in superplastic behavior at high deformation rates.

A decrease in deformation temperature leads to reduced elongation and non-uniform deformation, highlighting the inadvisability of lowering the deformation temperature below 725°C.



Figure 4. Tensile curves of alloys at a temperature of 750 °C and a strain rate of $2 \cdot 10^{-3} \text{ s}^{-1}$

Increasing the deformation temperature to 750°C leads to a significant decrease in the flow stress of the investigated alloys, which is explained by increased dislocation mobility and enhanced diffusion processes. However, along with this, a decrease in deformation uniformity is observed, which may be associated with uneven grain growth and the localization of plastic deformation in certain zones.

Nevertheless, the increase in temperature is also accompanied by a reduction in elongation, which is likely due to the intensive grain growth during deformation. At 750°C, grain boundaries become less stable, leading to grain coarsening, reduced grain boundary sliding, and deteriorated conditions for superplasticity. This effect is associated with a diminished ability of the material to maintain superplastic behavior at high temperatures, where dynamic recrystallization and grain coalescence begin to dominate. As a result, plasticity decreases, and the material becomes more prone to unstable flow, as confirmed by experimental data (Figure 4).

Table 1 presents the elongation test results of the alloys under various temperature-strain rate conditions.

The analysis of the data in Table 1 shows that the elongation of the alloys significantly depends on the temperature and strain rate. Thus, the optimal temperature-strain rate conditions for the 0Zr and 0.5Zr alloys are a temperature of 725°C and strain rates in the range of $2-5 \cdot 10^{-3}$ s⁻¹. For the 1Zr and 1.5Zr alloys, the optimal conditions are a temperature of 725°C and a strain rate of $5 \cdot 10^{-4}$ s⁻¹.

Table 2 presents data on the grain sizes of the α - and β phases, as well as the volume fraction of the β -phase in the alloys after superplastic deformation under various temperature-strain rate conditions. Table 1. Elongation values of alloys at different temperaturestrain rate conditions

| Alloy | Temperature, °C | Strain rate, c-1 | Elongation, % |
|------------------------------|---|--|---------------|
| | 700 | 2.10-3 | 280 |
| | | 5.10-4 | 450 |
| 0Zr | $\begin{array}{c c c c c c c c c c c c c c c c c c c $ | 490 | |
| | | $\begin{array}{r} \text{Strain rate, c}^{-1} \\ \hline 2\cdot 10^{-3} \\ \hline 5\cdot 10^{-4} \\ \hline 2\cdot 10^{-3} \\ \hline 5\cdot 10^{-3} \\ \hline 2\cdot 10^{-3} \\ \hline 2\cdot 10^{-3} \\ \hline 5\cdot 10^{-4} \\ \hline 2\cdot 10^{-3} \\ \hline 5\cdot 10^{-3} \\ \hline 2\cdot 10^{-3} \\ \hline 5\cdot 10^{-3} \\ \hline 2\cdot 10^{-3} \\ \hline 5\cdot 10^{-4} \\ \hline 2\cdot 10^{-3} \\ \hline 5\cdot 10^{-4} \\ \hline 2\cdot 10^{-3} \\ \hline 5\cdot 10^{-3} \\ \hline 2\cdot 10^{-3} \\ \hline 5\cdot 10^{-3} \\ \hline 2\cdot 10^{-3} \\ \hline 2\cdot$ | 580 |
| | 750 | | 380 |
| | 700 | 2.10-3 | 380 |
| 0.57. | $\begin{array}{c c} \mbox{Temperature, °C} & \mbox{Strain rate, c} \\ \hline 700 & 2 \cdot 10^{-3} \\ \hline 725 & 5 \cdot 10^{-4} \\ \hline 725 & 2 \cdot 10^{-3} \\ \hline 750 & 2 \cdot 10^{-3} \\ \hline 750 & 2 \cdot 10^{-3} \\ \hline 700 & 2 \cdot 10^{-3} \\ \hline 725 & 2 \cdot 10^{-3} \\ \hline 700 & 2 \cdot 10^{-3} \\ \hline 700 & 2 \cdot 10^{-3} \\ \hline 725 & 5 \cdot 10^{-4} \\ \hline 725 & 5 \cdot 10^{-4} \\ \hline 725 & 5 \cdot 10^{-3} \\ \hline 700 & 2 \cdot 10^{-3} \\ \hline 5 \cdot 10^{-3} \\ \hline 700 & 2 \cdot 10^{-3} \\ \hline 5 \cdot 10^{-3} \\ \hline 725 & 2 \cdot 10^{-3} \\ \hline 5 \cdot 10^{-3} \\ \hline 5 \cdot 10^{-3} \\ \hline 750 & 2 \cdot 10^{-3} \end{array}$ | 5.10-4 | 340 |
| 0Zr 0.5Zr 1Zr 1.5Zr | | 2.10-3 | 515 |
| | | $\begin{array}{c} 2\cdot 10^{-3} \\ \hline 5\cdot 10^{-4} \\ \hline 2\cdot 10^{-3} \\ \hline 5\cdot 10^{-3} \\ \hline 2\cdot 10^{-3} \\ \hline 5\cdot 10^{-4} \\ \hline 2\cdot 10^{-3} \\ \hline 5\cdot 10^{-3} \\ \hline 2\cdot 10^{-3} \\ \hline$ | 510 |
| | 700 | 2.10-3 | 450 |
| 17. | | 5.10-4 | 430 |
| IZr | 725 | $\begin{array}{r} {\rm Strain \ rate, \ c^{-1}}\\ \hline 2\cdot 10^{-3}\\ \hline 5\cdot 10^{-4}\\ \hline 2\cdot 10^{-3}\\ \hline 5\cdot 10^{-4}\\ \hline 2\cdot 10^{-3}\\ \hline 5\cdot 10^{-4}\\ \hline 2\cdot 10^{-3}\\ \hline 5\cdot 10^{-4}\\ \hline 2\cdot 10^{-3}\\ \hline 5\cdot 10^{-3}\\ \hline 2\cdot 10^{-3}\\ \hline 5\cdot 10^{-4}\\ \hline 2\cdot 10^{-3}\\ \hline 5\cdot 10^{-3}\\ \hline 2\cdot 10^{-3}\\ \hline 5\cdot 10^{-3}\\ \hline 2\cdot 10^{-3}\\ \hline \end{array}$ | 240 |
| | | 5.10-3 | 360 |
| 1.5Zr | 700 | 2.10-3 | 400 |
| | 725 | 5.10-4 | 550 |
| | | 2.10-3 | 360 |
| | | 5.10-3 | 350 |
| | 750 | 2.10-3 | 440 |

Table 2. Grain sizes of phases and volume fraction of β -phase in alloys after superplastic deformation under different conditions

| Alloy | Temperature, °C | Strain rate, c ⁻¹ | Grain size of the α- phase, μm | Grain size of the β-phase, μm | Volume fraction of the β-phase, % |
|-------|--------------------|---------------------------------|--------------------------------------|-------------------------------------|---|
| 0Zr | 725 | 2.10-3 | 1.7 ± 0.3 | 2.2 ± 0.3 | 56 |
| | | 5·10 ⁻³ | 1.6 ± 0.25 | $1.8 \pm 0,15$ | 46 |
| 0.5Zr | 700 | $2 \cdot 10^{-3}$ | 1.2 ± 0.1 | $1.5 \pm 0,\!15$ | 48 |
| | 725 | $2 \cdot 10^{-3}$ | 1.3 ± 0.1 | 1.75 ± 0.15 | 47 |
| | | 5·10 ⁻³ | 1.1 ± 0.1 | 1.6 ± 0.15 | 52 |
| 1Zr | 725 | 5·10 ⁻³ | 1.2 ± 0.1 | 1.5 ± 0.1 | 45 |
| | | 5.10-4 | 1.3 ± 0.1 | 1.9 ± 0.25 | 47 |
| 1.5Zr | 725 | 5·10 ⁻³ | 1.0 ± 0.1 | 1.55 ± 0.15 | 44 |
| | | 5.10-4 | 1.8 ± 0.1 | 2.3 ± 0.2 | 44 |
| | 750 | 2.10-3 | 1.4 ± 0.1 | 2.5 ± 0.25 | 63 |

As seen from Table 2, an increase in deformation temperature leads to a growth in the volume fraction of the β -phase. Additionally, the grain size of the α -phase remains almost unchanged under different testing conditions, whereas the grain size of the β -phase increases with decreasing strain rate and increasing deformation temperature.

To determine the mechanical properties of the alloys after superplastic deformation, tensile tests were conducted at a temperature of 725°C and a strain rate of $2 \cdot 10^{-3}$ s⁻¹ (Table 3). The samples were stretched to 100%, quenched in water, and then subjected to tensile testing at room temperature with a strain rate of 4 mm/min.

Table 3. Mechanical properties of alloys after 100% elongation at 725 °C and a strain rate of $2 \cdot 10^{-3} \text{ s}^{-1}$

| Alloy | Yield strength, MPa | Tensile strength, MPa | Elongation, % |
|-------|---------------------|-----------------------|---------------|
| 0Zr | 600 | 620 | 3 |
| 0.5Zr | 655 | 695 | 6 |
| 1Zr | 535 | 541 | 1 |
| 1.5Zr | 630 | 650 | 3 |

From the table, it is evident that the addition of zirconium enhances the strength characteristics of the alloys in both the initial and superplastic states. The highest yield strength and ultimate tensile strength values are observed in the 0.5Zr alloy, which also exhibits the greatest elongation, indicating an optimal combination of strength and ductility. However, a further increase in zirconium content leads to a decline in mechanical properties, likely due to grain growth and an increase in the volume fraction of the β -phase. In particular, the 1Zr alloy demonstrates the lowest values of yield strength, tensile strength, and elongation, indicating a significant reduction in ductility. To assess the impact of superplastic deformation on the alloy structure, microstructural studies were conducted using scanning electron microscopy (SEM). Figures 5 and 6 present the microstructures of alloys with different zirconium contents after deformation at 725°C with strain rates ranging from $5 \cdot 10^{-4}$ s⁻¹ to $2 \cdot 10^{-3}$ s⁻¹.









Figure 5. Microstructural images of the alloys after superplastic deformation at 725°C with a strain rate of $2 \cdot 10^{-3} \text{ s}^{-1}$: (a, b) – correspond to the 0Zr alloy, (c, d) – correspond to the 0.5Zr alloy,

Figure 6 shows the microstructure of alloys with different zirconium contents after superplastic deformation at 725°C and a strain rate of 2×10^{-3} s⁻¹. The alloy exhibits a fine-grained structure, characteristic of superplastic deformation, confirming the active development of dynamic recrystallization. The equiaxed grain shape indicates uniform strain distribution.



 SEM MAG: 10.0 kx
 WD: 9.09 mm
 Immediate
 VEGA3 TESC

 View field: 27.7 µm
 Det: BSE
 5 µm
 VEGA3 TESC





Figure 6. Microstructure images of the alloy after superplastic deformation at 725°C with a strain rate of $5 \times 10^{-4} \text{ s}^{-1}$: (a, b) – correspond to the 1Zr; (c, d) – correspond to the 1.5Zr

In zirconium-free alloys (0Zr), a homogeneous two-phase structure is observed, whereas the addition of 0.5% Zr leads to further grain refinement due to the slowing down of grain boundary diffusion.

In general, the structure of the alloys after superplastic deformation consists of a dispersed mixture of equiaxed grains of the α - and β -phases, indicating the development of dynamic recrystallization. Compared to the deformed state and annealing at similar temperatures, an increase in grain growth is observed due to the influence of high temperatures and prolonged plastic deformation. At the same time, the fine-grained structure is preserved due to the inhibition of grain growth by alloying elements such as zirconium. The presence of this structure positively affects the mechanical properties of the alloy, ensuring high plasticity and resistance to fracture. Thus, superplastic deformation has a significant impact on the microstructural characteristics of the alloy, determining its operational properties.

4. Conclusions

The conducted studies have established the effect of zirconium content on the superplastic behavior of titanium alloys under various temperature-strain rate conditions. Analysis of the obtained data showed that zirconium significantly influences the strain rate sensitivity coefficient (m), mechanical stability, and plasticity of the alloys, as well as the microstructural changes occurring during deformation.

It was found that at a temperature of 700°C, alloys containing zirconium exhibit a stable m coefficient value within a certain strain rate range (up to $2 \cdot 10^{-3} \text{ s}^{-1}$), after which a sharp decline is observed. In alloys without zirconium, the m coefficient decreases monotonously as the strain rate increases. At 725°C, the m coefficient exceeds 0.3 in all investigated alloys over a wide range of strain rates, indicating favorable conditions for superplastic flow. With a further increase in temperature to 750°C, the maximum m value (~0.35) is reached at a strain rate of $2 \cdot 10^{-3} \text{ s}^{-1}$; however, this is accompanied by grain coarsening, which negatively affects plasticity.

The optimal temperature-strain rate regimes for superplastic deformation were determined as follows:

– for alloys with 0% and 0.5% zirconium, the preferred temperature is 725°C, and the strain rate is in the range of 2– $5 \cdot 10^{-3} \text{ s}^{-1}$;

– for alloys with 1% and 1.5% zirconium, the optimal conditions include the same temperature (725°C) but a lower strain rate ($\sim 5 \cdot 10^{-4} \text{ s}^{-1}$).

All studied alloys demonstrated elongation of more than 200%; however, as the zirconium content increases above 0.5% and the temperature drops below 725°C, a decrease in plasticity and non-uniform deformation flow is observed, which may limit their application in superplastic processing. Additionally, an increase in zirconium content contributes to higher flow stress, which improves strength characteristics but reduces the material's ability to undergo uniform elongation. Meanwhile, increasing the temperature to 750°C reduces flow stress but is accompanied by grain coarsening, which deteriorates mechanical properties.

Microstructural analysis using scanning electron microscopy showed that after superplastic deformation, all alloys form a dispersed mixture of equiaxed grains of the α - and β phases. An increase in the average grain size compared to the initial state is observed, indicating processes of dynamic recovery and recrystallization. Alloys with higher zirconium content tend to form larger grains, which may reduce their technological plasticity during subsequent processing.

Thus, the research results confirm the feasibility of using the studied titanium alloys in superplastic forming processes under specific temperature-strain rate conditions. The identified patterns allow for the optimization of thermomechanical processing parameters aimed at achieving high plasticity and mechanical stability, which is essential for industrial applications, particularly in aerospace, space, and medical engineering. Further research may focus on studying the effects of additional alloying elements and pre-treatment conditions on the mechanical properties and microstructure of titanium alloys, which will expand their application in high-tech industries.

Author contributions

Conceptualization: A.M.A.; Data curation: A.M.A., A.Zh.T.; Formal analysis: A.M.A., R.A.Sh., G.M.K.; Funding acquisition: G.K.M., A.A.M.; Investigation: A.M.A., A.A.M.; Methodology: B.T.S., A.A.M.; Project administration: A.M.A., A.Zh.T.; Resources: A.A.M., B.T.S.; Software: B.T.S., A.A.M.; Supervision: G.M.K., G.K.M.; Validation: A.A.M., G.M.K.; Visualization: B.T.S., G.K.M.; Writing – original draft: A.M.A., G.M.K.; Writing – review & editing: A.M.A., A.A.M. All authors have read and agreed to the published version of the manuscript.

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Conflicts of interest

The authors declare no conflict of interest.

Data availability statement

The original contributions presented in this study are included in the article. Further inquiries can be directed to the corresponding author.

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Цирконийдің титан қорытпаларының құрылымы мен қасиеттерінің қалыптасуына әсері және оның аса пластикалық деформация кезіндегі рөлі

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Аңдатпа. Бұл жұмыста әртүрлі температуралар мен деформация жылдамдықтарында цирконийдің титан қорытпаларының аса пластикалық қасиеттеріне әсері зерттелді. Цирконийдің деформация жылдамдығына сезімталдық коэффициентіне (m), механикалық тұрақтылыққа және пластикалылыққа едәуір ықпал ететіні анықталды. Жоғары температура жағдайында цирконий бар қорытпалар белгілі бір деформация жылдамдықтарында m коэффициентінің тұрақты деңгейін көрсетеді, кейін ол күрт төмендейді. Ал цирконийсіз қорытпаларда деформация жылдамдығы артқан сайын бұл коэффициент біртіндеп азаяды. Аса пластикалық деформацияның оңтайлы температуралық және жылдамдықтық параметрлері цирконий мөлшеріне байланысты болады. Цирконийдің төмен концентрациясы бар қорытпалар орташа температура мен деформация жылдамдығында жоғары пластикалылықпен сипатталады, ал цирконийдің жоғары мөлшері бар қорытпалар біркелкі деформацияны қамтамасыз ету үшін төмен жылдамдықты талап етеді. Белгілі бір деңгейден асқан цирконий мөлшері пластикалылықтың төмендеуіне және деформацияның локализациясына әкеледі. Сонымен қатар цирконий аққыштық кернеуді арттырады, ал температураның көтерілуі бұл кернеуді азайтады, бірақ дәннің іріленуіне алып келеді, бұл механикалық қасиеттерге теріс әсер етеді. Сканерлейтін электрондық микроскопия әдісімен жүргізілген микроструктуралық талдау аса пластикалық деформациядан кейін барлық зерттелген қорытпаларда эквиаксы α- және β-дәндерден тұратын ұсақ дәнді құрылым қалыптасатынын көрсетті. Орташа дән өлшемі бастапқы күймен салыстырғанда артатыны анықталды, бұл динамикалық қалпына келу мен рекристаллизация процестерінің жүргенін білдіреді. Алынған нәтижелер зерттелген титан қорытпаларын аса пластикалық қалыптау технологияларында қолдануға болатының дәлелдейді. Айқындалған заңдылықтар бұл материалдардың өнеркәсіптік қолданылуы үшін жоғары пластикалылық пен механикалық тұрақтылықты қамтамасыз ететін оңтайлы термомеханикалық өңдеу параметрлерін анықтауға мүмкіндік береді.

Негізгі сөздер: ультра ұсақ дәнділік, қарқынды пластикалық деформация, құрылым, наноөлшемдер, аса пластикалылық, цирконий.

Роль циркония в формировании структуры и свойств титановых сплавов при сверхпластической деформации

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Аннотация. В работе исследовано влияние циркония на сверхпластические свойства титановых сплавов при различных температурах и скоростях деформации. Установлено, что цирконий оказывает значительное влияние на коэффициент чувствительности к скорости деформации (m), механическую стабильность и пластичность. В условиях повышенных температур сплавы, содержащие цирконий, демонстрируют устойчивый уровень коэффициента т в определённом диапазоне скоростей деформации, после чего происходит его резкое снижение. В сплавах без циркония наблюдается постепенное уменьшение коэффициента т с увеличением скорости деформации. Оптимальные температурно-скоростные параметры сверхпластической деформации зависят от содержания циркония. Сплавы с его низкой концентрацией характеризуются высокой пластичностью при умеренных температурах и средних скоростях деформации, тогда как сплавы с более высоким содержанием циркония требуют пониженных скоростей для обеспечения равномерности деформации. Увеличение содержания циркония выше определённого уровня приводит к снижению пластичности и локализации деформации. В то же время цирконий повышает напряжение течения, тогда как увеличение температуры способствует его снижению, но сопровождается укрупнением зерна, что отрицательно влияет на механические свойства. Микроструктурный анализ методом сканирующей электронной микроскопии показал, что после сверхпластической деформации все исследуемые сплавы формируют мелкозернистую структуру, состоящую из равноосных α- и β-зёрен. Установлено, что средний размер зерна увеличивается по сравнению с исходным состоянием, что свидетельствует о процессах динамического восстановления и рекристаллизации. Полученные результаты подтверждают возможность применения исследуемых титановых сплавов в технологиях сверхпластической формовки. Выявленные закономерности позволяют определить оптимальные параметры термомеханической обработки, обеспечивающие сочетание высокой пластичности и механической стабильности, что важно для промышленного использования данных материалов.

Ключевые слова: ультрамелкозернистость, интенсивная пластическая деформация, структура, наноразмеры, сверхпластичность, цирконий.

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