Hot deformation behavior and processing map of ultrafine-grained pure zirconium refined by compounding

Peisheng Han 1, Weijie Ma 1, Xiaoyu Zhu 1, Yanwei Li 1,2, XiRong Yang 1 and Xiaogang Wang 1,4

1 Shanxi Province Metallurgical Equipment Design Theory and Technology Key Laboratory (Provincial Department to Build National Key Laboratory Training Base), Taiyuan University of Science and Technology, Taiyuan 030024, People’s Republic of China
2 Metallurgical Engineering School, Xi’an University of Architecture and Technology, Xi’an 710055, People’s Republic of China
3 Mechanics School, Jinhong University, Jinhong 030619, People’s Republic of China
4 Authors to whom any correspondence should be addressed.

E-mail: 18334707506@163.com and wxg@tyust.edu.cn

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Abstract

An ultrafine-grained (UFG) pure zirconium (Zr) refined by compounding with a size of \( \Phi 4 \times 6 \) mm was subjected to a unidirectional compression test using a Gellble-3800 thermal simulation tester at the temperature of 300 °C–450 °C and a strain rate range of 0.001–0.05 s\(^{-1}\). Experimental results showed that the flow stress of UFG pure Zr refined by compounding is highly sensitive to temperature and strain rate, and the peak stress decreases with increase in deformation temperature and increases with increase in strain rate. The Arrhenius constitutive equation based on the experimental data can effectively predict peak stress in actual thermal deformation. The correlation coefficient between the actual value and the predicted value can reach as high as 0.99722. With the increase in deformation temperature and the decrease in strain rate, the UFG pure Zr refined by compounding otably undergoes dynamic recovery and dynamic recrystallization. These findings are based on the hot processing map and microstructure characteristics of UFG pure Zr refined by compounding. The optimal hot-working windows are determined to be in the deformation temperature of 320 °C–360 °C with strain rate of 0.005–0.01 s\(^{-1}\) and the deformation temperature of 410–450 °C with strain rate of 0.003–0.01 s\(^{-1}\).

1. Introduction

Pure zirconium (Zr) has good thermal conductivity, corrosion resistance, nuclear properties and good mechanical properties, and it is widely used in the atomic energy, chemical and biomedical industries [1, 2]. With the rapid development of the electronics industry and the precision machinery industry, the miniaturization of products has become a major trend in the industrial field. Industrial pure Zr, as an important structural metal, has a wide range of applications in micro-electro-mechanical systems and biomedical and aerospace fields [3–5]. However, industrial coarse-grained pure Zr has a grain size of approximately 23 \( \mu \)m, and the size effect is obvious in the field of metal plastic microformation in which the sample geometry is in the submillimeter level along the 2D direction [6, 7]. The surface quality of the part with Zr will be seriously affected by the size effect, and geometry and dimensional accuracy hardly meet the quality requirements of the products. By contrast, ultrafine-grain (UFG) materials are composed of uniform submicron grains, and have relatively higher strength, wear resistance, and corrosion resistance, and they can even produce superplastic deformation ability. UFG materials can improve the filling performance of microparts extremely well, indicating their important research significance in the field of plastic microformation [8, 9].

Various severe plastic deformation technologies have been successfully utilized in the preparation of UFG materials [10–13], and they primarily include equal channel angular pressing (ECAP), high-pressure torsion, and accumulated roll bonding. The rotary swaging technology can further refine the grain, make the structure...
After appearance of the peak stress, the material stress is gradually reduced. However, with the increase in strain rate, the stress gradually increases again. This is due to work hardening, the DRV and DRX exist simultaneously. Before the appearance of peak stress, the work stress peak appears in the curve, and then the curve trend decreases gradually. In the plastic deformation process, the interstitial atoms and vacancies are become increasingly active, and the dynamic softening causes the activated, and the kinetic energy of the metal atom is gradually increased. Moreover, the point defects, such as deformation temperature. The reason is that as the deformation temperature increases, a new slip system is activated, and thereby resulting in the tendency of the stress to level off and gradually increase. At the same strain rate, the flow stress of the UFG pure Zr increases with the increase in strain rate. The reason is that as the deformation rate increases, the material deformation rate increases, DRV and DRX cannot easily to occur, the internal dislocations and defects are further enhanced, and

| Table 1. Chemical composition of pure zirconium (ω/%). |
|----------------|---|---|---|---|---|---|
| Fe + Cr        | C  | N  | H  | O  | Zr |
| ≤0.2           | ≤0.05 | ≤0.025 | ≤0.005 | ≤0.16 | Bal. |

even, and can maximize the plasticity of material [14]. Therefore, in this study, the ECAP and rotary swaging composite deformation process is selected to successfully prepare the UFG pure Zr with a grain size of 200–250 nm. The existing studies on UFG materials have primarily focused on the microstructure evolution and mechanical properties of larger sample sizes. Yang [15], who conducted tensile experiments on UFG pure Zr, found that its yield strength and tensile strength can be significantly improved relative to those of coarse-grained pure Zr. Liu [16] studied the hot compression of UFG pure zirconium and found its strain rate sensitivity (m) to be in the range of 0.028–0.013, which is higher than that of coarse-grained pure zirconium. With the increase in strain temperature, the dynamic recovery (DRV) and dynamic recrystallization (DRX) of UFG pure Zr both become evident. However, the studies on the hot deformation behavior with of smaller sample size and low strain rates are few.

The strength of composite deformed UFG pure Zr is high, but using a microforming equipment is difficult when performing forming experiments at room temperature, thus, thermoforming is usually selected [17]. In this study, the hot compression experiments for the UFG pure Zr at different temperatures and rates were carried out with a Gellble-3800 thermomechanical simulator, allowing the researchers to obtain the true stress–true strain curve. Accordingly, the Arrhenius hot-deformation equation with Z parameters is determined by calculating the derived experimental data. Then, the energy dissipation and plastic instability maps are drawn, and the two maps are superimposed to obtain the hot-processing map of the composite deformation of the UFG pure Zr. The microstructure of the compressed samples is observed by transmission electron microscopy (TEM), Subsequently, the hot-deformation rule of the UFG pure Zr is analyzed, and its reliability is verified. The results can provide a theoretical basis for the development and optimization of the plastic microforming process.

2. Experiment

A hot-rolled annealed industrial pure Zr sheet was used for the experiment, and its chemical composition (ω/%) are shown in table 1. The UFG pure Zr was successfully obtained by optimizing the ECAP-prepared samples by the rotary swaging process. The wire on the swaging bar was cut to obtain a sample with a size of φ4 × 6 mm, and a hot-compression experiment was performed using a Gellble-3800 thermomechanical simulator. The deformation temperatures were set to 300 °C, 350 °C, 400 °C, 450 °C, while the strain rates were 0.001, 0.005, 0.010, 0.050 s⁻¹. The maximum deformation was 50%, while the heating rate was 5 °C / s⁻¹. A Special lubricating oil and a graphite sheet were used to reduce the influence of friction on the end face of the sample. The microstructure of the sample was retained by performing water cooling immediately after hot compression, and the sample was cut parallel to the compression direction. Then, the microstructure was observed by high-resolution TEM (JEM-200CX) and scanning electron microscopy (SEM).

3. Results and discussion

3.1. True stress–strain curve

Figure 1 shows the true stress-strain curve of the UFG pure Zr with different strain rates. As the strain increases, a stress peak appears in the curve, and then the curve trend decreases gradually. In the plastic deformation process, the work hardening, the DRV and DRX exist simultaneously. Before the appearance of peak stress, the work hardening rate is significantly larger than the DRV and DRX rates, and the flow stress increased significantly. After appearance of the peak stress, the material’s DRX is enhanced, the DRX softening becomes evident, and the stress is gradually reduced. However, with the increase in strain rate, the stress gradually increases again. This phenomenon can be explained by the friction between the specimen and the end face of the fixture that increases with the increase in deformation, thereby resulting in the tendency of the stress to level off and gradually increase. At the same strain rate, the flow stress of the UFG pure Zr is decreased remarkably with the increase in deformation temperature. The reason is that as the deformation temperature increases, a new slip system is activated, and the kinetic energy of the metal atom is gradually increased. Moreover, the point defects, such as the interstitial atoms and vacancies, are become increasingly active, and the dynamic softening causes the flow stress to decrease. At the same deformation temperature, the flow stress of the UFG pure Zr increases with the increase in strain rate. The reason is that as the deformation rate increases, the material deformation rate increases, DRV and DRX cannot easily to occur, the internal dislocations and defects are further enhanced, and
additional dislocation entanglements occur inside the material, thereby increasing stress. When strain rate is high, the UFG pure zirconium undergoes work hardening, and the peak stress is reached. A large deformation rate indicates that the material deformation time is short, the internal energy generated in a short time cannot be transferred to the surrounding medium, resulting in a rapid increase in internal energy, a significant dynamic softening, and a significant decrease in flow stress. Comparatively, a lower deformation temperature means a greater softening effect of the material. Figures 1(c) and (d) show the true stress-strain curves of the deformation temperature at 300 °C.

3.2. Arrhenius constitutive model
The hot deformation of the metal is a heat-activated process. The influence of deformation rate and temperature on flow stress during hot deformation can be expressed by the Arrhenius constitutive equation [18], as follows:

\[
\dot{\varepsilon} = K f(\sigma) \exp \left( \frac{-Q}{RT} \right) \tag{1}
\]

The relationship between strain rate and deformation temperature in the hot deformation can be expressed by the Zener–Hollomon parameter (Z), as follows:

\[
Z = \dot{\varepsilon} \exp \left( \frac{Q}{RT} \right) \tag{2}
\]

\[
f(\sigma) = \begin{cases} 
\sigma^n \alpha \sigma & \text{if } \alpha \sigma \leq 0.8 \\
\exp (\beta \sigma) & \text{if } \alpha \sigma \geq 1.2 \\
[\sinh (\alpha \sigma)]^{n'} & \text{all } \sigma 
\end{cases} \tag{3}
\]

where \( K \) is the material parameter, \( f(\sigma) \) is a flow stress function, \( Q \) is the activation energy of the material’s hot deformation (\( J/\text{mol} \)), \( R \) is the constant (8.314 \( J \text{ mol}^{-1} \text{ K}^{-1} \)), \( T \) is the absolute temperature, \( \alpha \) and \( \beta \) are the stress multiples, \( n \) and \( n' \) are stress indices, and \( \alpha = \beta / n' \).

By substituting all the stress cases \( f(\sigma) \) in equations (1) and (3) with equation (2), the relationship function between \( Z \) and \( \sigma \) is obtained as follows:
By taking the natural logarithm of the two sides of equation (4), the flow stress can be written as a functional relationship as:

$$
\ln [\sinh (\alpha \sigma)] = \frac{1}{n} \ln Z - \frac{1}{n} \ln K
$$

and

$$
\sigma = \frac{1}{\alpha} \ln \left\{ \left( \frac{Z}{K} \right)^{\frac{1}{n}} + \left[ \left( \frac{Z}{K} \right)^{\frac{1}{n}} + 1 \right]^2 \right\}.
$$

In equation (6), we use the peak stress data obtained in figure 1 to determine the $\alpha$, $n$, $Q$ and $K$ values. For the case of low ($\alpha \sigma \leq 0.8$) and high stress levels ($\alpha \sigma \geq 1.2$), the corresponding $f(\sigma)$ values are substituted with equation (1), and the following relationship can be obtained:

$$
\begin{cases}
\dot{\varepsilon} = K' \sigma' & \alpha \sigma \leq 0.8 \\
\dot{\varepsilon} = K'' \exp (\beta \sigma) & \alpha \sigma \geq 1.2
\end{cases}
$$

where $K'$ and $K''$ are material parameters at a given deformation temperature. By taking the natural logarithm of the two sides of equation (7), the following formula can be obtained:

$$
\begin{cases}
\ln \sigma = \frac{1}{n'} \ln \dot{\varepsilon} - \frac{1}{n'} \ln K' & \alpha \sigma \leq 0.8 \\
\sigma = \frac{1}{\beta} \ln \dot{\varepsilon} - \frac{1}{\beta} \ln K'' & \alpha \sigma \geq 1.2
\end{cases}
$$

The experimental peak stress and the corresponding strain rate substituted with equation (8) obtain the relationship between $\ln \sigma - \ln \dot{\varepsilon}$ and $\sigma - \ln \dot{\varepsilon}$ at different deformation temperatures. The result is shown in figure 2.

On the basis of the average value of the slopes of each set of straight lines, $n'$ and $\beta$ can be obtained, and the $\alpha$ value can be calculated.

For all stress levels, substituting $f(\sigma) = [\sinh (\alpha \sigma)]^n$ with equation (1) results in the following expression:

$$
\dot{\varepsilon} = K [\sinh (\alpha \sigma)]^n \exp \left( -\frac{Q}{RT} \right).
$$

The natural logarithms of both sides of equation (9) can be obtained as follows:

$$
\ln [\sinh (\alpha \sigma)] = \frac{Q}{nRT} - \frac{1}{n} \ln k + \frac{1}{n} \ln \dot{\varepsilon}.
$$

The experimental peak flow stress data substituted with equation (10) for the plotting of all the slopes of $\ln [\sinh (\alpha \sigma)] - \ln \dot{\varepsilon}$ at the different deformation temperatures (figure 3(a)) and then the $n$ value is obtained. Figure 3(b) shows the plot all slopes of $\ln [\sinh (\alpha \sigma)] - 1/T$ at the different strain rates, which are used to obtain the $Q$ value (figure 3(b)).
Substituting the obtained $Q$ with equation (4) derives the Zener–Hollomon parameter corresponding to the peak strain for the different temperatures and strain rates. The obtained $n$ value is further substituted with equation (5) to obtain the fitting relationship of $\ln Z = \ln \left[ \sinh(\alpha \sigma) \right]$, as shown in figure 4 below. The $K$ value can be obtained from the intercept of the linearly fitted line $\ln Z = \ln \left[ \sinh(\alpha \sigma) \right]$. The hot-deformation parametric values are shown in table 2.

Substituting all the parameters with equation (6) can obtain the hot-deformation equation of the peak stress of the UFG pure Zr for the different temperatures and strain rates.

$$\sigma = \frac{1}{0.002951} \ln \left\{ \left( \frac{z}{1.822 \times 10^6} \right)^{\frac{1}{15.42} + \frac{1}{1.822 \times 10^6}} + 1 \right\}$$

(11)

**Table 2.** Parametric values of hot deformation.

| $n'$ | $\beta$ | $\alpha$ | $n$ | $Q(\text{K/mol})$ | $K$ |
|------|---------|---------|-----|-----------------|-----|
| 20.42 | 0.060243 | 0.002951 | 15.42 | 105.8 | $1.822 \times 10^6$ |

**Figure 3.** Linear fit relationship of (a) $\ln \left[ \sinh(\alpha \sigma) \right] - \ln \dot{\varepsilon}$; and (b) $\ln \left[ \sinh(\alpha \sigma) \right] - 1/T$.

**Figure 4.** Linear fit relationship of $\ln Z = \ln \left[ \sinh(\alpha \sigma) \right]$. 
The experimentally obtained peak stress data for the different strain rates and temperatures are substituted with equation (11) to verify the accuracy of the Arrhenius constitutive model. The comparison between the calculated values of the Arrhenius constitutive model and the experimental data is shown in figure 5. According to the results, the Arrhenius constitutive equation has a high correlation coefficient (0.99722) and a low average relative error (5.9220%), indicating that the Arrhenius constitutive equation has a good accuracy in terms of predicting the macroscopic peak stress.

### 3.3. Establishment and analysis of the hot-processing map

A hot-processing map provides an important basis for optimizing the hot processing of materials and controlling the evolution of materials. Hot-processing maps can directly reflect the deformation law of materials under different deformation temperatures and strain rates, evaluate the possible instability of materials in the hot-forming process, and provide the optimal hot-processing route [19, 20]. At present, the most widely used hot-deformation model is the dynamic material mechanics model proposed by Prasad et al [21]. This model can effectively describe the relationship between plastic deformation and structural dissipation and clarify how the external energy diffuses through the plastic deformation of the workpiece. Moreover, the model considers the deformation process as a closed thermodynamic system, the total energy P inputted to the workpiece during the deformation process is transformed into two parts dissipation amount (G) and dissipation coordination (J). The relationship is given by

\[
P = \sigma \dot{\varepsilon} = G + J = \int_0^\infty \sigma d\varepsilon + \int_0^\sigma \varepsilon d\sigma.
\]

The distribution ratio of G and J can be expressed by m, and the corresponding relationship is given by

\[
m = \frac{J}{G} = \frac{\partial (\log \sigma)}{\partial (\log \varepsilon)}.
\]

When the deformation temperature and strain are constant, the stress and strain rates of the specimen and m are regarded to be constitutively correlated. The relationship is given by

\[
\sigma = K_0 \dot{\varepsilon}^m, \quad \varepsilon = K_1 \sigma^{1/m} \quad (\text{K and } K_0 \text{ are constants}),
\]

Substituting equation (14) with equation (13) obtains the definition of J, as follows:

\[
J = \int_0^\infty \left( \frac{\sigma}{K} \right)^{\frac{1}{m}} d\sigma = \frac{m}{m + 1} \sigma \dot{\varepsilon}.
\]

When \( m = 1 \), the material is in an ideal dissipative state. At this time, \( J \) reaches the maximum of \( J_{\text{max}} = 0.5 \sigma \dot{\varepsilon} \).

Therefore, the ratio of the actual J to the maximum J (\( J_{\text{max}} \)) can be used to obtain the efficiency of power dissipation (\( \eta \)) of the material. The relationship can be described as follows:

![Figure 5. Comparison between the prediction value of the Arrhenius constitutive equation and experimental value.](image)
The \( \eta \) value can reflect the hot-processing performance of the materials to a certain extent. However, a higher \( \eta \) value is not necessarily good, it is also related to the instability region. To judge the instability of materials, Prasad et al.\(^\text{[22]}\) proposed the inequality criterion for plastic instability as follows:

\[

\xi (\dot{\varepsilon}) = \frac{\partial \ln \left( \frac{m}{m+1} \right)}{\partial \ln \dot{\varepsilon}} + m < 0.
\]

where \( \xi (\dot{\varepsilon}) \) is a constant, thereby reflecting the function of the strain rate and the deformation temperature. With this criterion, the plastic instability map of the material can be drawn on a 2D plane, and the hot-processing map of the material can be obtained by superimposing the efficiency map of power dissipation and the instability map.

The hot-processing map of the UFG pure Zr is shown in figure 6. From the hot-processing map, the efficiency of power dissipation, the processing safety zone and the instability region can be observed. The strain rate and the temperature variation range of the suitable material processing are obtained, and the instability region is avoided as much as possible.

Figure 6 shows the hot-processing map of the UFG pure Zr. The values on the contour line correspond to the power dissipation efficiency, the white area is the processing safety zone, and the gray area is the instability zone. As shown by the figure, the power dissipation efficiency decreases with the increase in strain rate. At the low strain rate, the deformation temperature is approximately 340 °C, and the value of the power dissipation efficiency reaches the maximum. However, the power dissipation efficiency does not change remarkably with the increase in deformation temperature because the growth of the high temperature grains reduces the value of the power dissipation efficiency\(^\text{[23]}\). The coupling effect of DRX and grain growth causes the deformation temperature to change minimally for the value of the power dissipation efficiency.

Plastic instability occurs in the region of the relatively high strain rate (domains 5 and 6). This result is observed when the strain rate is high, and the internal temperature of the material does not have sufficient time to dissipate into the surrounding medium due to the short deformation time. Thus, the temperature is increased, resulting in the hot softening of materials to form adiabatic shear bands\(^\text{[24]}\), and further causing material deformation instability. Plastic instability also occurs at the low temperature and for the strain rate range of 0.002–0.005 s\(^{-1}\). Further investigation and analysis are carried out subsequently in combination with the microstructure. According to the above analysis, when the deformation temperature ranges from 330 °C to 360 °C, and the strain rate is 0.001 s\(^{-1}\) (domain 1), the power dissipation efficiency is high. However, the controllable strain rate range is narrow, avoiding the instability region in actual production becomes difficult, and the processing efficiency becomes low. Therefore, the optimum hot-processing area is selected as in two domains follows: deformation temperature of 320 °C–360 °C with strain rate of 0.005–0.01 s\(^{-1}\) (domain 3), and deformation temperature of 410–450 °C with strain rate of 0.003–0.01 s\(^{-1}\) (domain 4).
3.4. Microstructural observation and analysis

Figure 7(a) is shows the TEM image of the UFG pure Zr after two passes of ECAP deformation and rotary swaging optimization, while figures 7(b)–(e) show the TEM images of the microstructure after compression deformation at different deformation temperatures and the strain rate of 0.005 s⁻¹.

When the deformation temperature is 300 °C, many dislocations and dislocation cells are present inside the structure, and the material’s DRV form a small amount of subgrains (figure 7(b)). When the deformation temperature is 350 °C, the intragranular dislocation density gradually decreases, and the grain boundaries become clear (figure 7(c)). The subgrains are combined to form a large-angle subcrystal with a much larger size and a sharp interface, and the material changes from DRV to DRX. When the deformation temperature increases to 400 °C (figure 7(d)), many subgrain boundaries disappear due to subgrain consolidation, and the subgrains grow into much larger angular grains with clearer grain boundaries and an evident DRX. When the temperature rises to 450 °C (figure 7(e)), the grain grows to the maximum, and the dislocation density is extremely low, the work hardening effect is eliminated or partially eliminated during the hot deformation. The flow stress gradually decreases with the increase in temperature. As shown in the above figures, with the increase in deformation temperature, the DRV and DRX of the materials become significant, and the machining safety

![Figure 7. TEM structure before and after deformation of the UFG pure Zr. (a) as-ECAPed (b) T = 300 °C (c) T = 350 °C (d) T = 400 °C (e) T = 450 °C.](image-url)
zone exists in the region in which a fully dynamic DRX occurs, (figure 6; domain 4). The results are consistent with the true stress–true strain curves and the hot-processing map above.

Figure 8 shows the TEM images the UFG pure Zr after compression with different strain rates at the deformation temperature of 450 °C. As the strain rate increased, the DRX grain size gradually decreases, and an incompleteness is observed. When the deformation temperature is 300 °C and the strain rate is 0.05 s⁻¹ (figure 6; domain 5), as shown in figure 8(d), the incomplete DRX phenomenon becomes remarkable. The reason is that when the strain rate is high, the deformation speed is fast, the deformation time is short, and several recrystallized grains have no time to grow, thereby resulting in grain refinement [25], DRX is not completely carried out, and the microstructure appears with underdeveloped large and small crystal grains, resulting in uneven deformation and plastic instability. When the deformation temperature is 450 °C and the strain rate is 0.001 s⁻¹ (figure 6; domain 7), as shown in figure 8(a), the grains in this region becomes coarse, the mixed crystal phenomenon becomes serious, the instability phenomenon becomes evident, and the processing property of the material becomes poor.

Figure 9 shows the SEM images of the UFG pure Zr after compression at the deformation temperature of 300 °C and strain rate of 0.005 s⁻¹ (domain 2). A clear crack appears (figure 9(a)), and few microscopic holes are observed around the surrounding (figure 9(b)). As the deformation increased, the micropores gradually develop, and the enlarged holes join together to form cracks, thereby causing a plastic instability of the material.

4. Conclusions

1. The flow stress of UFG pure Zr with increased strain during hot deformation gradually decreases after reaching the peak stress, as the strain is further increased, the flow stress also increases. The flow stress is extremely sensitive to temperature and strain rate. The peak stress decreases with the increase of deformation temperature and the decrease of strain rate.
2. The Arrhenius constitutive equation is established on the basis of the peak stress of the composite deformation of the UFG pure Zr. The hot-deformation activation energy of the material is given by

\[ Q = 105.81 \text{ KJ mol}^{-1} \]

3. According to the hot-processing map of the UFG pure Zr, at the deformation temperature of 320 °C–360 °C with strain rate of 0.005–0.01 s\(^{-1}\) (domain 3), and the deformation temperature of 410–450 °C with strain rate of 0.003–0.01 s\(^{-1}\). DRX fully occurs, which corresponds to a safe processing region. Local instability is primarily distributed at the low-temperature low-strain rate and high temperature high-strain rate region.

4. On the basis of the microstructural analysis for the different regions, the UFG pure Zr has evident DRV and DRX at the deformation temperature ranging within of 400 °C–450 °C and strain rate of 0.001–0.005 s\(^{-1}\). The flow characteristics of the materials and the accuracy of the hot-processing map have been verified effectively.

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ORCID iDs

Peisheng Han @ https://orcid.org/0000-0001-8162-7929
Yanwei Li @ https://orcid.org/0000-0002-6899-2259

References

[1] Li X J 2015 Progress in the titanium industry 01 44
[2] Xiao D W, Li Y L and Hu S S 2008 Rare Metal Materials and Engineering 37 2122–4
[3] Engd U and Eckstein R 2002 Journal of Materials Processing Technology 125–126 55–44
[4] Vollertsen F, Niehoff H S and Hu Z 2006 International Journal of Machine Tools and Manufacture 46 1172–9
[5] Zheng W, Song L B and Wang G C 2011 Materials Science and Technology 19 7–8
[6] Raulea L V, Goijaerts A M and Govaert L E 2001 Journal of Materials Processing Technology 115 44–8
[7] Vollertsen F, Biermann D and Hansen H N 2009 CIRP Annals—Manufacturing Technology 58 566–87
[8] Valiev R Z and Langdon T G 2006 Progress in Materials Science 51 881–981
[9] Chang C.C. and Kuo W L 2010 Proc. of the Institution of Mechanical Engineers, Part B: Journal of Engineering Manufacture 224, 1767–73
[10] Terada D, Inoue S and Tsuji N 2007 Journal of Materials Science 42 1673–81
[11] Shirouyeh M, Xu J and Langdon T G 2014 Materials Science and Engineering A 614 223–31
[12] Yzou G Y and Huang M N 2000 Journal of Materials Processing Technology 105 344–51
[13] Zhao X, Yang X and Liu X 2014 Materials Science and Engineering: A 607 482–9
[14] Fang S M, Lei T and Zhang Y L 2007 Forging & Stamping Technology 32 69–72
[15] Yang X R, Zhang W Y and Liu X Y 2019 Rare Metal Materials and Engineering 48 178–83
[16] Liu X Y, Yang C and Luo L 2019 Chinese Journal Of Rare Metals 38 500–6
[17] Wang X G, Liu J L and Zhao X C 2017 Journal of Wuhan University of Technology-Mater. Sci. Ed 32 437–43
[18] Sellars C M and McEgart W J 1966 Acta Metallurgica 14 1136–8

Figure 9. SEM images of the UFG pure Zr after compression at the deformation temperature of 300 °C and strain rate of 0.005 s\(^{-1}\).
[19] Prasad Y V R K 2003 Journal of Materials Engineering and Performance 12 638–45
[20] Zhang J L, Guo H Z and Liang H Q 2016 Rare Metals 35 118–26
[21] Prasad Y V R K 1996 Metallurgical & Materials Transactions A 27 235–6
[22] Prasad Y V R K 1990 Indian Journal of Technology 28 435–51
[23] Wang Z, Sun R and Song G 2012 Special Casting & Nonferrous Alloys 9 55–7
[24] Zener C and Hollomon J H 1944 Journal of Applied Physics 15 22–32
[25] Liu X Y, Zhao X C and Yang X R 2012 Rare Metal Materials and Engineering 41 667–71