

Article

The Effect of Cooling Rate on Microstructure and Mechanical Properties of the Zr-4Hf-3Nb (wt%) Alloy

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Abstract: The mechanical properties of Zr-based alloys, such as strength and elongation, are heavily dependent on the cooling rate during heat treatment. Understanding the phase transformation and microstructural evolution in various cooling media can establish the connection between the cooling rate and mechanical properties. The effect of the cooling rate on the phase, microstructure, and tensile properties of Zr-4Hf-3Nb (wt%) alloy is studied in this paper. The results show that the phase composition of the samples transforms from $\alpha+\beta$ to $\alpha+\beta+\omega$, and, finally, to $\alpha+\alpha'+\omega$, while the average grain size of α phase decreases from 3.73 μm to 1.96 μm , and the distribution varies from compact to scattering as the cooling rate increases. Hf tends to distribute in β phase, and the slower cooling rate is beneficial to the existence of Hf. The strength and microhardness enhances monotonously, while the elongation ascends first, then decreases as the cooling rate increases. The high strength of water-cooling samples is attributed to the reduction in average grain size and volume fraction of α phase, and the lath α' martensite and granular ω phase. The fracture pattern of Zr-4Hf-3Nb (wt%) alloy is ductile fracture, and the plasticity gets better with decreasing cooling rate.



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Keywords: cooling rate; Zr-4Hf-3Nb (wt%) alloy; phase; microstructure; mechanical properties

1. Introduction

The demand of corrosion resistant structures in chemical and nuclear industries drives the research and development of zirconium alloys. As an important anti-corrosion material, zirconium alloy possesses the advantages of excellent corrosion resistance and low thermal neutron absorption cross-section [1–6]. Zr-4Hf-3Nb (wt%) alloy has been widely used in the environment of alternating acid and alkali, high temperature and pressure, such as pressure vessel for chemical industry, washing tower chassis, tray support, and cladding tube for nuclear reactor [7,8]. In order to prolong the component replacement cycle, the methods used to improve the strength of metallic materials include adding alloy elements to the base metal [9–11], rolling deformation treatment to change the microstructure [12,13], adjusting alloy composition to precipitate the second phase [14,15], refining the grain size to the appropriate degree [16,17], etc. These methods enhance the strength by hindering the movement of dislocations in the way of introducing defects in microstructures but damage the plasticity and toughness concurrently.

It is well known that the mechanical properties of metal materials are significantly affected by the microstructure. Additionally, the heat treatment is an effective method to control the microstructure [18], which can change the morphology, size, distribution, and volume fraction of the constituent phase. Additionally, a conventional approach for determining the heat treatment conditions is to adjust the cooling rate [19–21]. In recent years, some scholars have studied the phase transformation, microstructural evolution, and mechanical properties of zirconium alloys during cooling. Zhang et al. [22] studied

the effect of cooling process on the formation of duplex microstructure in Zr-2.3Nb alloy, and clarified that when the cooling rate increased from furnace cooling to water cooling, both the grain nucleation rate and growth rate increased, and the microstructure changed from equiaxed to duplex. Kulkarni et al. [23] investigated the mechanical properties of Zr-2.5Nb pressure tube material subjected to heat treatments in $\alpha+\beta$ phase field, and reported that when the cooling rate increased to 50 °C/s, the grain size decreased, and the ultimate tensile strength increased to 750 MPa. Dong et al. [18] studied the microstructural evolution, mechanical properties, and corrosion behaviors using cooling rate regulation in Zr-40Ti-4.5Al-4.5V alloy, and revealed that when the cooling rate decreased from air cooling to furnace cooling, the alloy took place the phase transformation of $\beta \rightarrow \alpha + \alpha''$ martensite, and the number of slip systems reduced, resulting in the ultimate tensile strength up to 1462 MPa. Hsu et al. [24] studied the effects of heat treatments on the structure and mechanical properties of Zr-30Ti alloys, and found that due to the precipitation of ω phase, the brittle fracture occurred in furnace cooling sample, leading to the low bending strength of 600 MPa. At present, the research focused on the influence of cooling rate on phase, microstructure, and tensile properties of zirconium alloy mainly focuses on Zr-Nb and Zr-Ti alloy. There are few studies on Zr-4Hf-3Nb (wt%) alloy, which has been developed with good corrosion resistance, making it a potential high performance metallic material. Nevertheless, the difference in the processing step will alter the phase and the microstructure, and further affect the mechanical properties. Therefore, it is necessary to control the microstructural evolution and mechanical properties of Zr-4Hf-3Nb (wt%) alloy by adjusting the cooling rate during heat treatment.

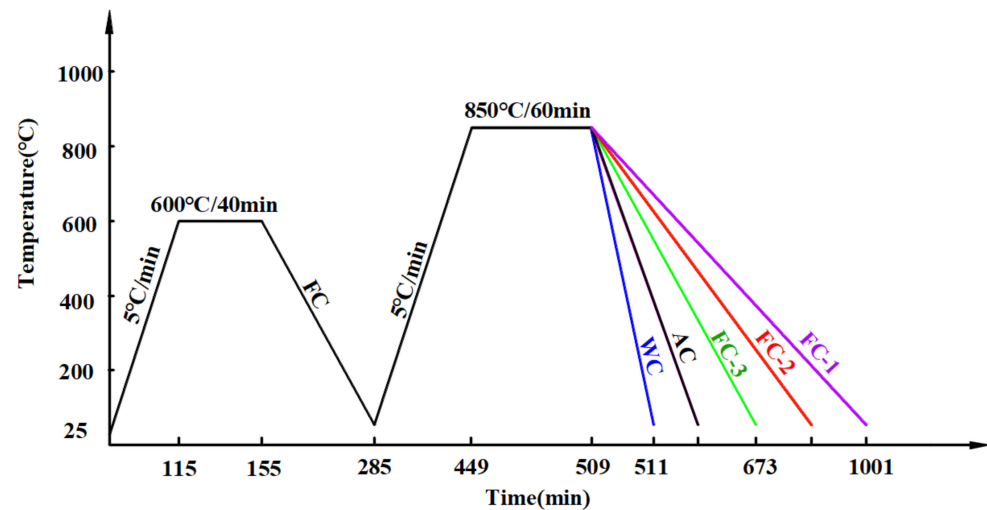
In the present work, the effect of cooling rate on phase transformation, microstructural evolution, and mechanical properties of Zr-4Hf-3Nb (wt%) alloy is systematically studied. The X-ray diffraction patterns (XRD), optical microscope (OM), and scanning electron microscopy-energy dispersive spectrometry (SEM-EDS) and transmission electron microscopy (TEM) are used to observe the morphology and phase composition of microstructures. Corresponding mechanical responses are investigated by using Vickers microhardness test and tensile test at room temperature. Achievements in this study will play an important role in the optimization of performance for Zr-4Hf-3Nb (wt%) alloy. This work provides a theoretical basis for the development of Zr-4Hf-3Nb (wt%) alloy design and theoretical models.

2. Experimental

Typical Zr-4Hf-3Nb (wt%) alloy plates with the diameter of 10 mm and length of 20 mm were used in this study. The chemical composition of the alloy was listed in Table 1, and the content of Hf and Nb was 4.47% and 3.01%, respectively. To relieve the residual stress, the samples were homogenized at 5 °C/min to 600 °C for 40 min in a vacuum tube furnace and consequently cooled to room temperature. Subsequently, the samples were reheated at 5 °C/min to 850 °C within the $\alpha+\beta$ phase field for 60 min, then cooled to room temperature with different cooling rate, including three kinds of furnace cooling with distinct cooling time (named as FC-1, FC-2, and FC-3, respectively), air cooling (AC) and water cooling (WC). FC-1, FC-2 and FC-3 referred to cooling in the furnace with various cooling time after heat preservation. AC referred to putting the sample in the medium of air and cooling to room temperature after heat preservation. WC referred to putting the sample in the medium of water and cooling to room temperature after heat preservation. Figure 1 shows the schematic diagram of the heat treatment processing route. During the cooling process from 850 °C to room temperature, the change of temperature was recorded every 5 min to calculate the cooling rate of each time period, and the average values. The cooling rate of FC-1, FC-2, FC-3, AC, and WC samples were approximately 0.05, 0.10, 0.15, 5 and 1000 °C/s, respectively.

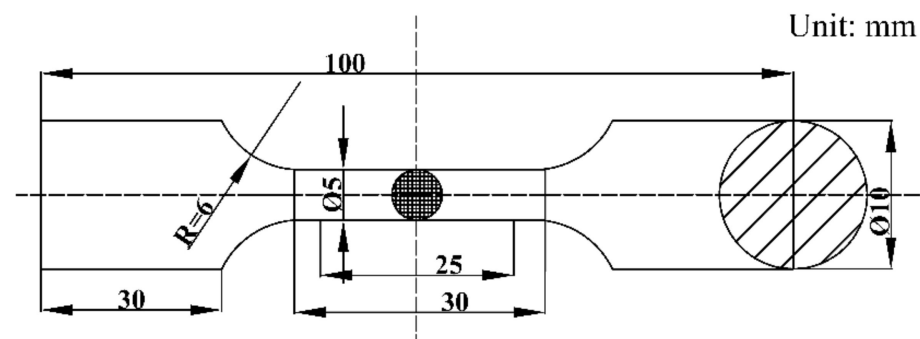
Table 1. Chemical composition of Zr-4Hf-3Nb (wt%) alloy.

Element	Zr	Nb	Hf	Fe	Ti
wt%	92.27	3.01	4.47	0.16	0.09

**Figure 1.** The schematic diagram of the heat treatment processing route.

The heat-treated samples were sliced using a diamond wheel cut off machine to obtain for XRD, OM, SEM-EDS, and TEM. The OM and SEM samples were first polished, then etched using a mixture of 5 vol.% hydrofluoric acid, 45 vol.% nitric acid, and 50 vol.% distilled water. The TEM samples were prepared by using twin-jet electro-polishing in a solution containing 85 vol% methanol, 15 vol% perchloric at -30°C , and 15 V.

Tensile tests were conducted at room temperature using a CMT-100 testing machine at a strain rate of $5 \times 10^{-4} \text{ s}^{-1}$. Figure 2 shows the dimensions of the bone-shaped plate sample with the diameter of 10 mm and length of 100 mm in tensile test. Vickers microhardness measurements were performed on a Q30 Microhardness Tester after the samples were polished. A load of 200 g was applied for 10 s at each location. Fracture surface morphologies of the failed samples were examined using SEM to characterize the crack propagation mode. There were 25 test samples in total, of which 5 samples were taken from each cooling rate, and 5 areas were selected for microhardness test for each sample. The total number of measurement points was 125 to ensure the accuracy of the test results.

**Figure 2.** The schematic diagram of the tensile sample with two-dimensional dimension.

3. Results and Discussion

3.1. Phase Composition Analysis

Zr alloys had two kinds of crystal structure, namely, high-temperature β phase with body centered cubic structure and low-temperature α phase with hexagonal close-packed

structure. During the cooling process, the β phase transformed into α phase at around 865 °C. When Zr alloys were quenched from a two-phase or β single phase region, three types of martensite phases can appear in sequence as the β phase stability increased, including α' phase with hexagonal close-packed, α'' phase with orthorhombic, and ω phase with distorted hexagonal close-packed structure [25].

According to the study of Yafei Pan, the Zr-Hf phase diagram is shown in Figure 3, and exhibited the phases liquid, β -Hf, β -Zr, and α -Hf, α -Zr. There was no intermediate compound in the binary system.

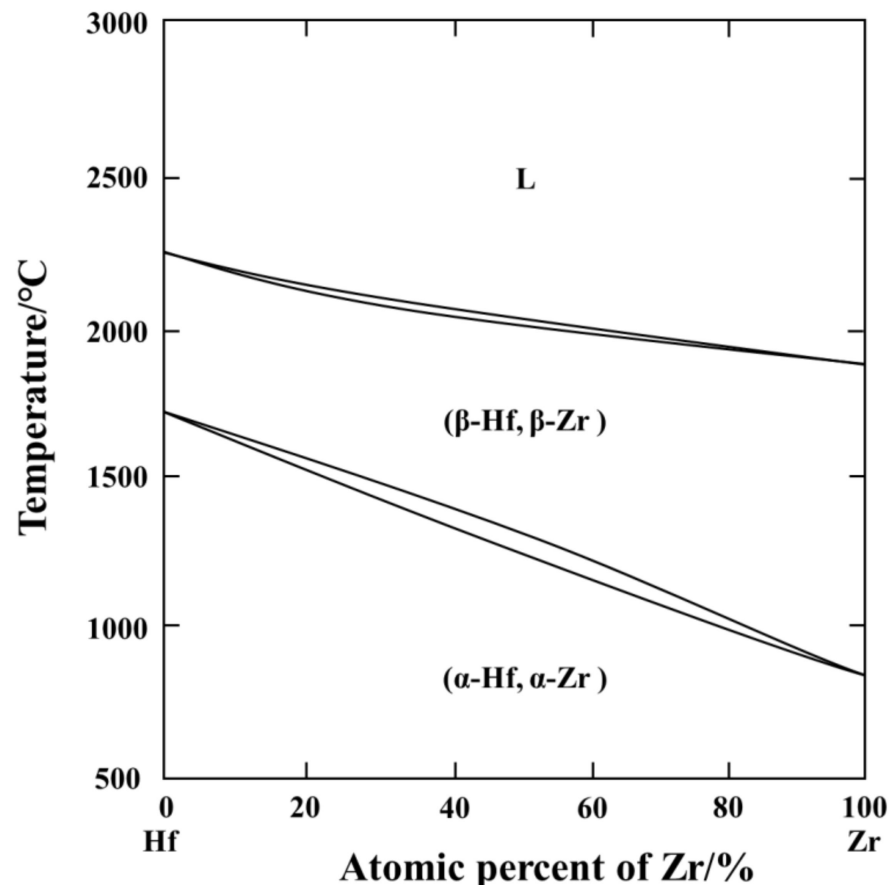


Figure 3. The Zr-Hf phase diagram.

Figure 4 shows the XRD patterns of Zr-4Hf-3Nb (wt%) alloy samples under different cooling rate. Since all the samples were heat treated in $\alpha+\beta$ region and the α phase remained stable throughout the cooling process, they all presented the diffraction peak of α phase after cooling. In furnace cooling process, the samples mainly contained the α phase and a small amount of the β phase. With the increase in cooling rate from FC-1 to FC-3, the diffraction peak intensities at $2\theta = 31.9^\circ$, 34.8° , and 73.5° , respectively, decreased, indicating the phase transformation of $\beta \rightarrow \alpha$ was restrained. When the cooling rate increased in AC, the sample mainly showed the α and β phase. Additionally, in contrast to the FC-3 sample, the intensity of α phase peaks further reduced. In addition, there was a small peak at $2\theta = 35.9^\circ$, and it was identified to be ω phase. When the cooling rate increased in WC, the sample contained α and ω phase. The intensity of α phase peaks at $2\theta = 34.8^\circ$ enhanced rapidly, and the diffraction peak intensity of ω phase also increased.

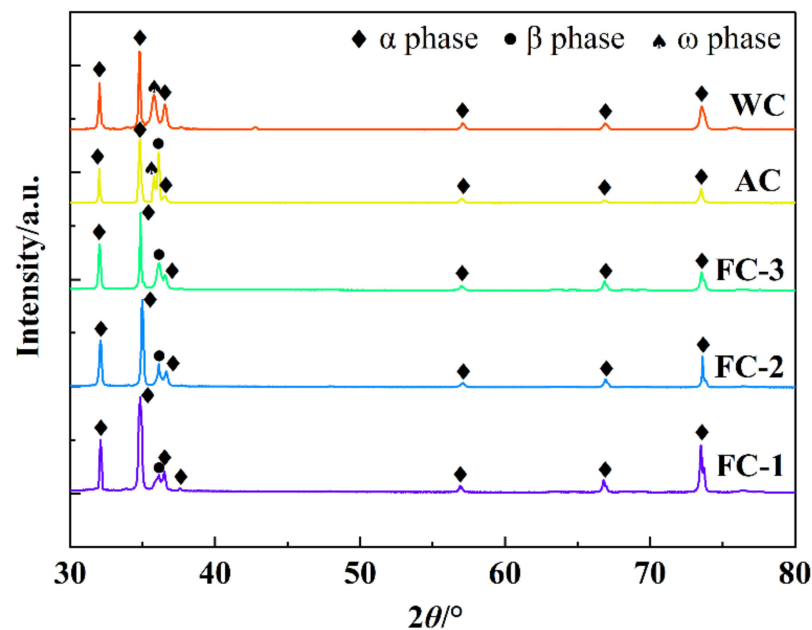


Figure 4. XRD patterns of Zr-4Hf-3Nb (wt%) alloy with various cooling rate.

Due to the slow cooling rate of furnace cooling process, the phase transformation of $\beta \rightarrow \alpha$ conducted smoothly, forming quite a few α phases. As the cooling rate increased in AC, the process of $\beta \rightarrow \alpha$ was subjected to some resistance, β phase tended to transform to be ω phase instead of α phase. As the cooling rate enhanced to WC, the high cooling rate conducted to the occurrence of $\beta \rightarrow \omega$, promoting the precipitation of ω phase.

3.2. Microstructure Analysis

Figure 5 illustrated the OM micrographs and grain size distribution histograms of Zr-4Hf-3Nb (wt%) alloy under different cooling rate. The light white part was α phase, using Photoshop software to adjust the contrast of α phase, then the Image Pro Plus software was selected to calculate the average grain size and volume fraction of α phase.

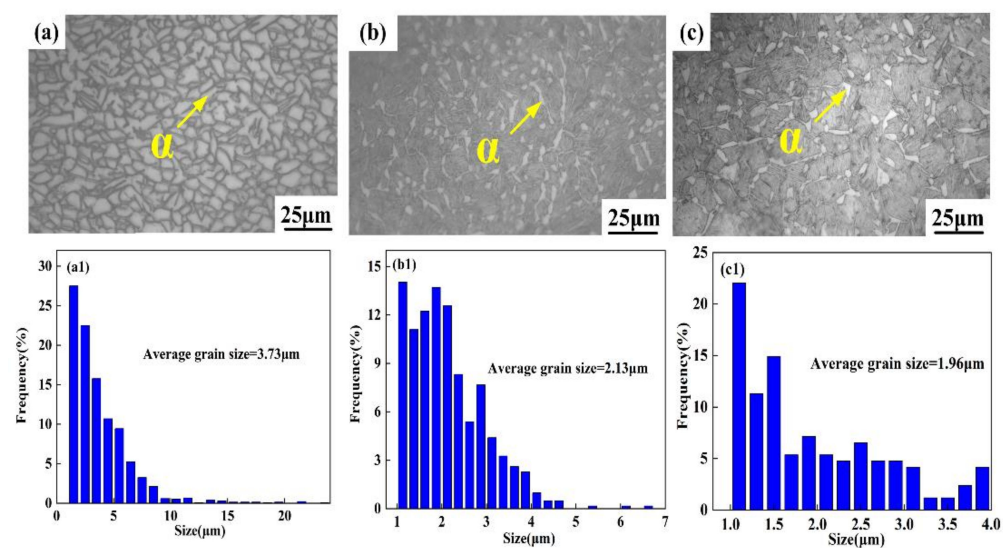


Figure 5. OM micrographs and grain size distribution histograms of Zr-4Hf-3Nb (wt%) alloy with various cooling rate: (a,a1) FC-1, (b,b1) AC, (c,c1) WC.

As shown in Figure 5, when the cooling rate increased from FC-1 to AC, then to WC, the average grain size of α phase decreased from 3.73 μm to 2.13 μm , then to 1.96 μm . The

grain size of α phase mainly depended on the nucleation rate and growth velocity during crystallization. With increasing growth velocity, the α phase of the FC-1 sample presented the coarse grain size of 3.73 μm . As the nucleation rate increased, the quantity of grains enhanced, and the α phase of the WC sample exhibited the fine grain size of 1.96 μm . When the cooling rate enhanced from FC-1 to WC, the nucleation rate enhanced faster than the growth velocity with the enlargement of the undercooling degree, leading to the grain refinement of α phase by 47.45%.

The volume fraction of α phase with different cooling rate was showed in Table 2. As the cooling rate increased from FC-1 to FC-3, then to WC, the volume fraction of α phase first decreased slightly, then dropped significantly and finally stabilized at 11.37%. Cooling rate was an important factor to control the phase transition of metal materials during heat treatment. Although all the samples were heat treated in $\alpha+\beta$ region, the low cooling rate induced higher content of α phase, and the high cooling rate promoted the process of $\beta \rightarrow \omega$.

Table 2. The volume fraction of α phase in Zr-4Hf-3Nb (wt%) alloy at different cooling rate.

Samples	FC-1	FC-2	FC-3	AC	WC
Volume fraction of α phase	91.91%	85.94%	76.26%	13.60%	11.37%

Figure 6 showed the SEM images of the alloy samples at different cooling rate. In furnace cooling process, the equiaxed grains in α phase were dominant in the microstructure, and the residual β phase located at the interface between α phases. With increasing cooling rate, the distribution of residual β phase varied from dispersive to continuous network-like. In AC sample, there were a small amount of bulk primary α phase (α_p) retained from the $\alpha+\beta$ two-phase zone and a few secondary α phase (α_s) with lath structure precipitated from β phase. In WC sample, apart from the bulk α_p phase, there was a new phase, α' martensite with regular fine lath structure and crisscross distribution, which could not be observed in the XRD. The high cooling rate completely suppressed the transition of $\beta \rightarrow \alpha$, and the phase transformation of β to α' occurred, which was a metastable state in the process of β to α phase. However, the α' martensite would be retained due to the inadequate thermodynamic conditions of time and energy.

Table 3 shows the results of the EDS analysis in the electron microscope for the FC-1 and AC sample. It could be seen that the content of Hf in α phase (5.11%, 5.00%) was lower than the one in β phase (11.96%, 8.26%) for the FC-1 and AC sample, respectively. In addition, the content of Hf in β phase for the FC-1 sample was higher than the AC sample, which demonstrated that Hf tended to distribute in β phase, and the slower cooling rate was beneficial to the existence of Hf. The α phase of the FC-1 sample obtained sufficient growth by the migration pattern due to the long stay at high temperature of 850 $^{\circ}\text{C}$ compared to the AC sample, making the α phase grow fully to be bulk. When the sample rapidly cooled to room temperature by AC, there were the phase transformations of $\beta \rightarrow \alpha$ and $\beta \rightarrow \omega$, forming the small and lamellar α phase. That was to say, the cooling rate affected the proportion of α and β phase in the microstructure and influenced the content of Hf in α and β phase.

Figure 7 shows the results of EDS elemental mapping analysis for the WC sample in Figure 6e. It could be seen that Zr and Nb mainly filled the matrix, and the distribution of Hf and Ti was homogeneous. Compared with the element of Ti and Nb, the content of Hf was low. In other words, the high cooling rate was not conducive to the existence of Hf.

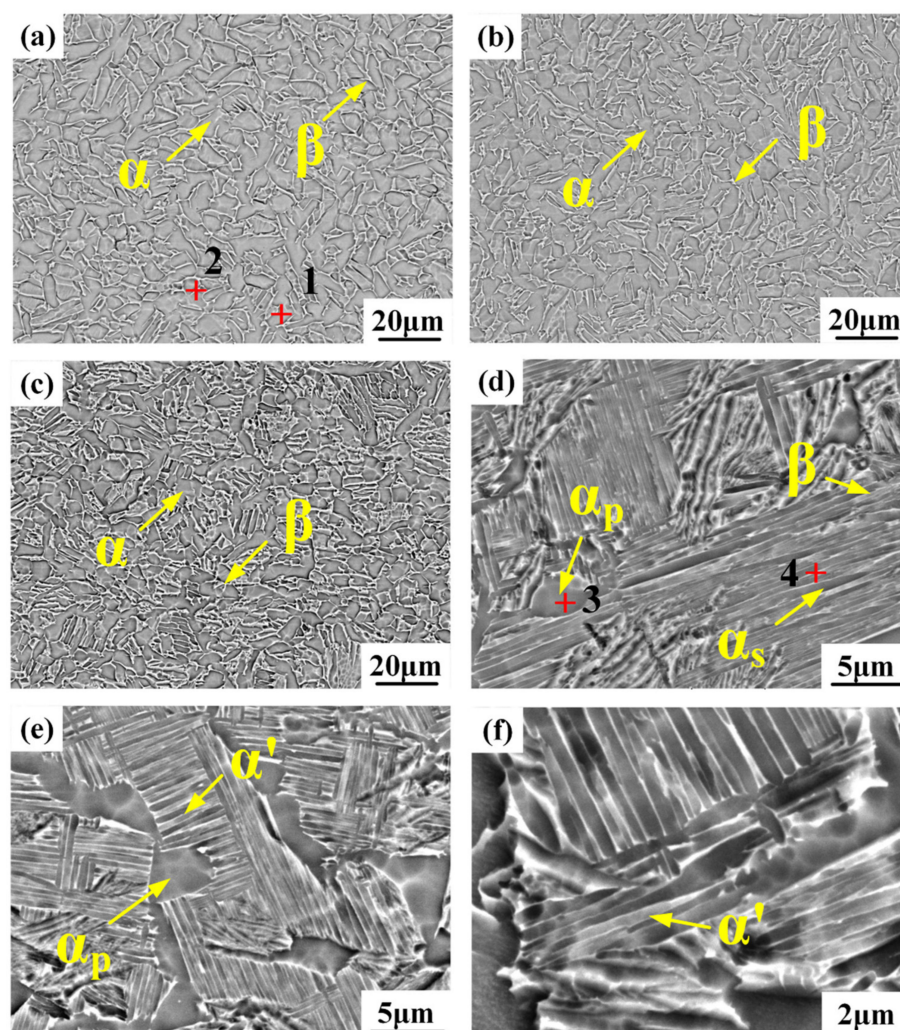


Figure 6. SEM micrographs of Zr-4Hf-3Nb (wt%) alloy with various cooling rate: (a) FC-1, (b) FC-2, (c) FC-3, (d) AC, (e,f) WC.

Table 3. EDS analysis of the position 1, 2, 3, and 4 in Figure 6.

Cooling Rate	Phase	Position	Element	Weight/%	Atomic/%
FC-1	α	1	Zr	91.91	93.29
			Hf	5.11	5.09
			Nb	2.92	1.52
			Ti	0.06	0.10
			Total	100	100
	β	2	Zr	85.71	86.66
			Hf	11.96	11.87
			Nb	2.15	1.11
			Ti	0.18	0.36
AC	α	3	Total	100	100
			Zr	92.31	93.24
			Hf	5.00	4.96
			Nb	2.38	1.23
			Ti	0.32	0.57
	β	4	Total	100	100
			Zr	89.38	90.56
			Hf	8.26	8.22
			Nb	2.07	0.91
			Ti	0.29	0.31
			Total	100	100

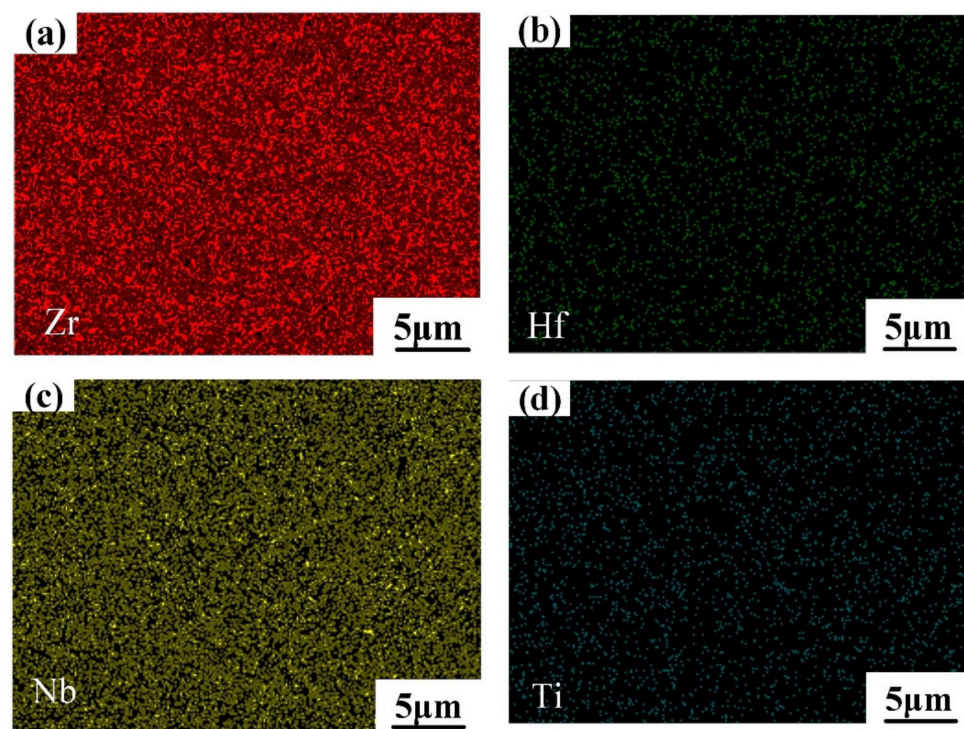


Figure 7. The EDS mapping of Zr (a), Hf (b), Nb (c), and Ti (d) of the samples with WC in Figure 6e.

Figure 8 shows the bright-field TEM micrographs and corresponding selected area electron diffraction (SAED) patterns of constituent phase in Zr-4Hf-3Nb (wt%) alloy samples with different cooling rate. Figure 8a–c depicted a typical bulk α phase with hexagonal close packed (hcp) structure, and the corresponding crystal orientation were $[001]$, $[11\bar{1}]$, and $[00\bar{1}]$ in furnace cooling process. A small amount of β phase located at the boundary between α phases. From Figure 8d, it could be seen that the thick lath α_s phase with hexagonal close packed (hcp) structure was presented in AC sample, and the corresponding crystal orientation was $[00\bar{1}]$. In WC sample, there were a few lath α' martensite with staggered distribution because of the phase transformation of β to α' martensite induced by the high cooling rate. In addition, the thickness of α' martensite ($0.18\ \mu\text{m}$) was narrow than the α_s phase ($0.39\ \mu\text{m}$) in AC sample, which was contributed to increasing the strength of the WC sample. Additionally, there were dislocations within the lath α' martensite, as shown in Figure 8f. The existence of dislocations could improve the hardness and strength of Zr-4Hf-3Nb (wt%) alloy owing to dislocation strengthening.

Figure 9 shows the dark-field TEM images of the AC and WC sample. The fine granular morphology could be observed, and it was identified to be the ω phase. Compared to the AC sample, the distribution of ω phase in WC sample was uniform and the quantity was a little more. It was consistent with the result in the XRD that the diffraction peak intensity of ω phase in AC sample was lower than the WC sample.

3.3. Mechanical Properties Analysis

3.3.1. Tensile Properties

The engineering stress versus strain curves of Zr-4Hf-3Nb (wt%) alloy for all cooling conditions are displayed in Figure 10, especially for the WC sample accompanied by the ultimate tensile strength (UTS) and total elongation (TE) of 618 MPa and 16.09%, respectively. Compared to the WC sample, the samples in furnace cooling process exhibited lower UTS, but higher TE. Figure 11 represents the variation of strength and elongation with cooling rate. As shown in Figure 11, with increasing cooling rate, the YS and UTS increased from 383 MPa and 432 MPa to 536 MPa and 618 MPa, respectively. However, the

elongation first enhanced to 21.02% for the FC-2 sample, then decreased to 16.09% for the WC sample.

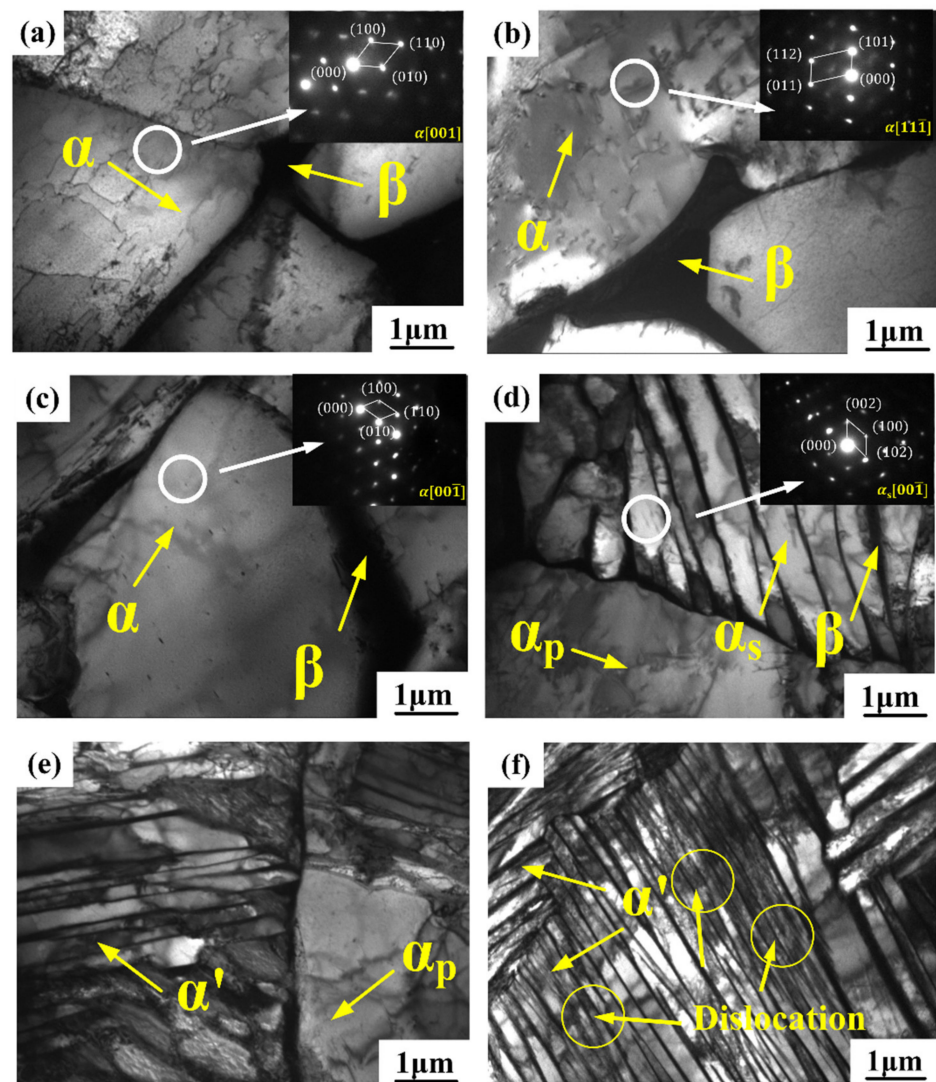


Figure 8. The bright-field TEM images and corresponding SAED patterns of Zr-4Hf-3Nb (wt%) alloy with various cooling rate: (a) FC-1, (b) FC-2, (c) FC-3, (d) AC, (e,f) WC.

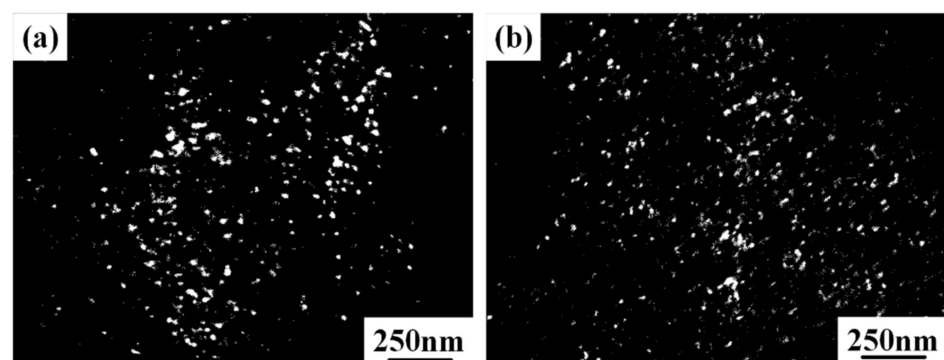


Figure 9. The dark-field TEM images revealing the fine ω particles in the Zr-4Hf-3Nb (wt%) alloy. All the bright spots are fine ω particles. (a) AC; (b) WC.

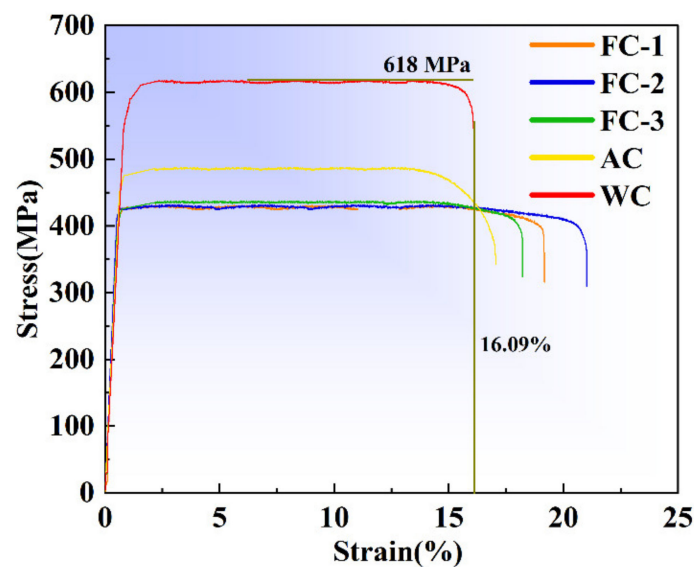


Figure 10. Engineering stress-strain curves of Zr-4Hf-3Nb (wt%) alloy. With various cooling rate.

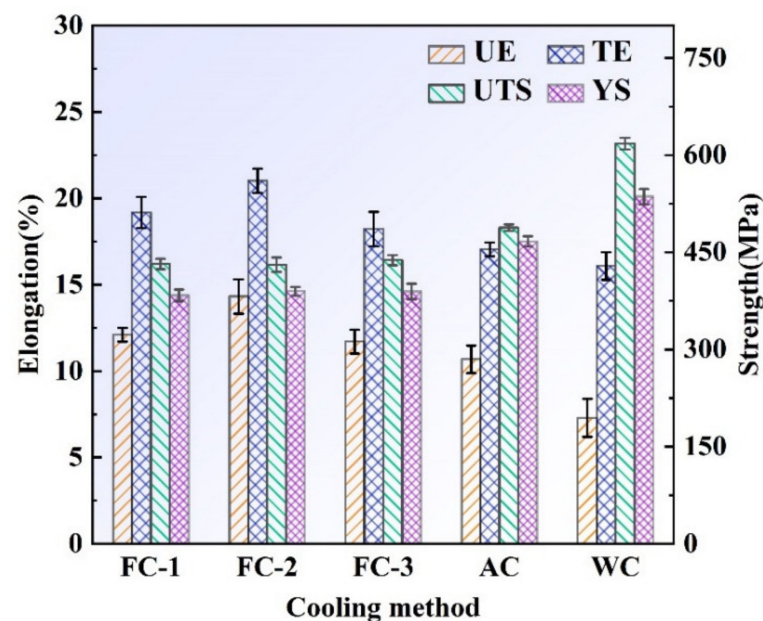


Figure 11. The variation of strength and elongation of Zr-4Hf-3Nb (wt%) alloy. with various cooling rate.

The mechanical properties of Zr-4Hf-3Nb (wt%) alloy were affected by the microstructural evolution such as morphology, size, distribution, and volume fraction of constituent phase, which were significantly dependent on the cooling rate. With increasing cooling rate, the tensile properties of Zr-4Hf-3Nb (wt%) alloy altered evidently, including a rapid enhancement in strength and a nonmonotonic decrease in elongation. When the cooling rate increased from FC-1 to FC-3, the samples had nearly the same and moderate values of YS and UTS, which could be ascribed to the similar morphology and distribution of lath α and residual β phase. The slow cooling rate led to the diffusion-controlled growth of α phase and the low tensile strength in Zr-4Hf-3Nb (wt%) alloy. In the AC and WC sample, the metastable ω phase caused the enhancement of strength and the decrease in ductility. The increasing cooling rate reduced the grain size and added the grain boundary area, which enhanced the movement resistance of dislocation in lath α' martensite and led to the high strength of the WC sample. In the tensile process, elastic deformation appeared first with the increase of load. When the value enhanced beyond the elastic limit of material,

uniform plastic deformation occurred, and the elongation in this stage was called uniform elongation (UE). When the value of tensile stress per unit area exceeded the UTS, the alloy necked and fractured, and the elongation corresponded to the non-uniform elongation. The sum of uniform and non-uniform elongation equaled to the total elongation (TE) [26,27]. When the cooling rate increased from FC-1 to FC-2, the grain size of α phase decreased from 3.73 μm to 3.28 μm . Additionally, the stress distributed homogeneously during the tensile test, weakening the stress concentration. Therefore, the FC-2 sample exhibited the better TE of 21.02%. However, the elongation decreased from FC-2 to WC, which was consistent with the law that the higher the YS, the lower the elongation, since the local stress concentration led to the premature fracture and reduced elasticity of the alloy.

Figure 12 shows the variation of static toughness (U_T), yield ratio (YR) and the product of strength and elongation (PSE) with various cooling rate. The U_T shall be considered as the total area under the stress-strain curve, which was an indication of the energy per unit volume that can be absorbed by the material without fracture. It could be seen from Figure 12a that the U_T of the FC-1, FC-3, and AC samples were almost the same (nearly 6400 MJ/m³), while the U_T of the FC-2 sample was the highest (7370 MJ/m³), which implied the FC-2 sample possessed a good combination of strength and plasticity.

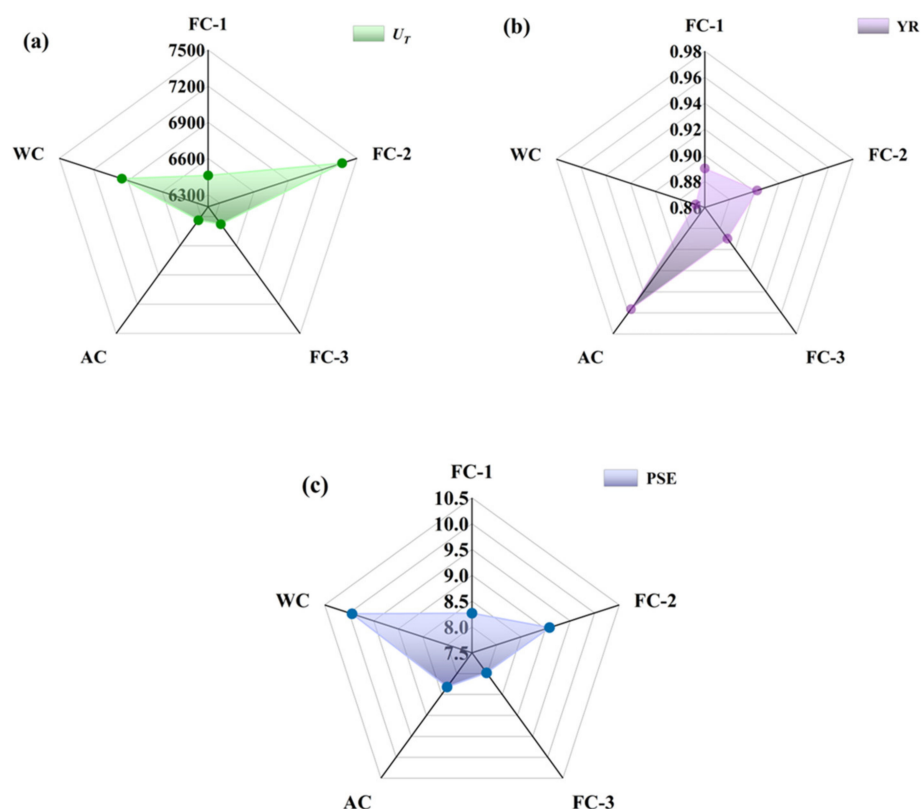


Figure 12. The variation of U_T (a), YR (b), PSE (c) of Zr-4Hf-3Nb (wt%) alloy. with various cooling rate.

The smaller grain size of α phase in FC-2 sample made the stress distribution uniform and reduced the stress concentration, then the sample had a higher TE and presented a better U_T . However, the WC sample had a lower TE due to the existence of ω phase, which can worsen the plasticity; thus, it exhibited an inferior U_T . Compared with Zr-2.3Nb alloy, the U_T of Zr-4Hf-3Nb (wt%) alloy was lower. This can be attributed to the fact that Zr-2.3Nb alloy showed a duplex microstructure, while Zr-4Hf-3Nb (wt%) alloy exhibited an equiaxed microstructure.

The YR referred to the proportion of yield strength to tensile strength of materials, which can be regarded as a coefficient to measure the strength reserve of materials. From

Figure 12b, the AC sample showed a higher YR of 0.96 than others, revealing that it exhibited a weaker strengthening effect but maintained high strength during the deformation process. The higher the value of YR, the lower the strain hardening capacity. As the short duration of the yield stage, the UTS of AC sample were close to YS, leading to the high YR. Due to the existence of α' martensite and ω phase in WC sample, the deformation degree was more serious, which made the UTS higher, and, thus, the YR lower.

The product of strength and elongation (PSE) was thought to be an index of ability to absorb the impact energy and can be approximated by the equation of $PSE \approx \sigma \varepsilon$, where σ was the UTS and ε was the elongation. As depicted in Figure 12c, the PSE of the FC-1 and AC sample were nearly the same (about 8.3 GPa·%), while the WC sample presented the higher PSE value of 9.94 GPa·%, which showed the WC sample had the stronger ability to assimilate the impact energy. A large amount of α' martensite, ω phase and the dislocation in WC sample significantly improved the strength while reducing the plasticity, and the comprehensive result was that the WC sample presented an excellent PSE. Compared with Zr-2.3Nb alloy [22], the PSE of Zr-4Hf-3Nb (wt%) alloy was lower. This is due to the Zr-2.3Nb alloy conducted a plastic deformation of 55% than the Zr-4Hf-3Nb (wt%) alloy before heat treatment.

3.3.2. Microhardness

Figure 13 presented the Vickers microhardness and corresponding microstructural evolution of Zr-4Hf-3Nb (wt%) alloy with different cooling rate. It was obvious that the microhardness kept in pace with increasing cooling rate, which was consistent with the law that Vickers microhardness was positively related to the tensile strength for metallic materials. Compared to the FC-1 sample of 162.98 HV, the Vickers microhardness of the FC-2 sample slightly increased to 173.7 HV, which could be ascribed to the reduction in the grain size of α phase. The refined lath structure of α_p phase in the AC sample and α' martensite in the WC sample enhanced the microhardness evidently.

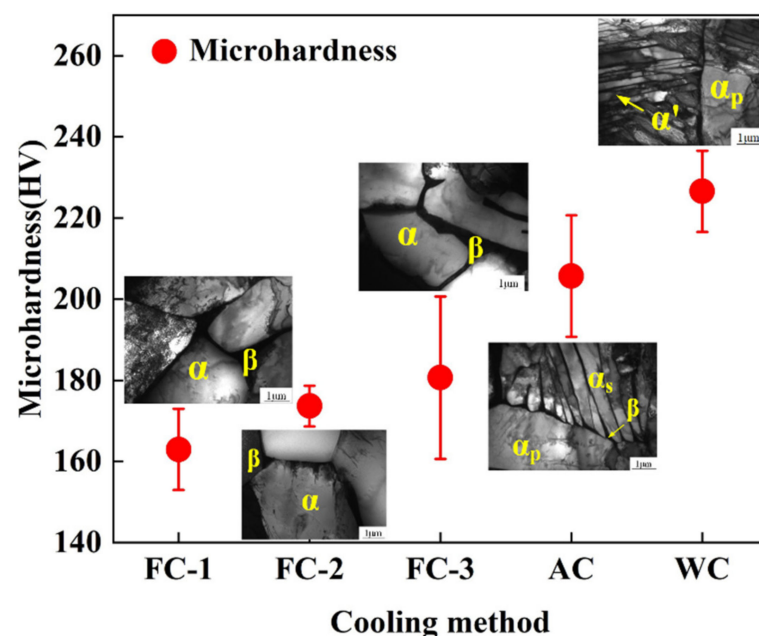


Figure 13. The Vickers microhardness of Zr-4Hf-3Nb (wt%) alloy. with various cooling rate.

3.4. Fracture Characteristics

Figure 14 shows the SEM images of fracture surface in Zr-4Hf-3Nb (wt%) alloy samples with different cooling rate. The ductile dimples were predominant in the fractures of all samples, indicating that the tension crack propagation occurred in a ductile fracture mode. The fracture surface of the FC-2 sample consisted of large and deep dimples, implying

splendid plasticity. However, similar characteristics with small and shallow dimples were observed in WC sample. The reduction in energy absorbed by fracture led to the decrease in static toughness of the WC sample, as shown in Figure 12a.

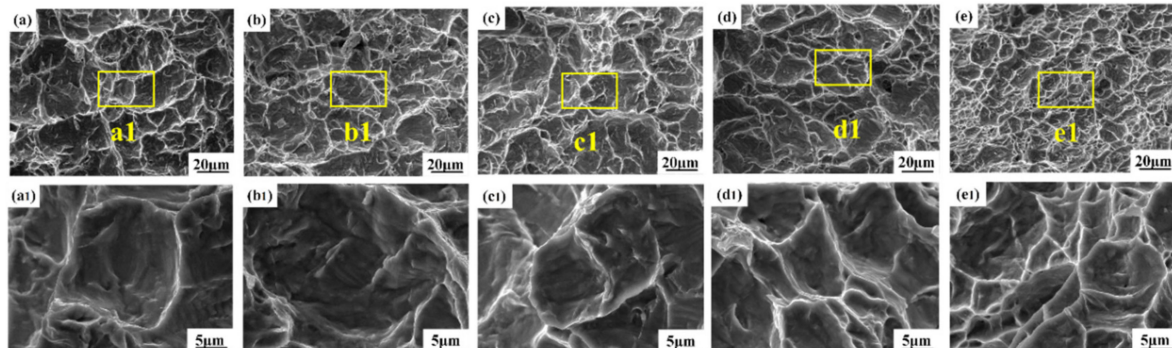


Figure 14. Morphology of the fracture surface of Zr-4Hf-3Nb (wt%) alloy. with various cooling rate: (a,a1) FC-1, (b,b1) FC-2, (c,c1) FC-3, (d,d1) AC, (e,e1) WC.

Figure 15 shows the dimple size distribution of fractographies in Zr-4Hf-3Nb (wt%) alloy with different cooling rate based on the quantitative analysis. It could be seen that the size of ductile dimples gradually decreased as the cooling rate increased. The FC-2 sample presented the larger dimple size of 28.5 μm . However, the WC sample exhibited the smaller dimple size of 8.08 μm . The larger size of dimples in samples with lower cooling rate showed that more energy was needed for failure under deformation.

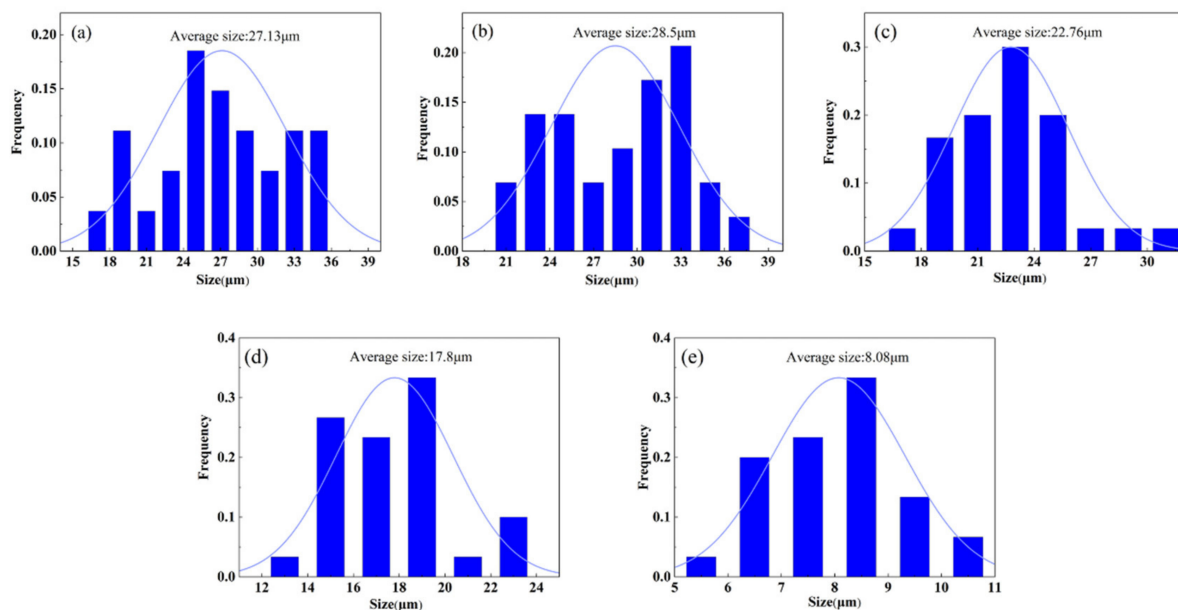


Figure 15. The dimple size distribution histograms of fractographies in Zr-4Hf-3Nb (wt%) alloy with various cooling rate: (a) FC-1, (b) FC-2, (c) FC-3, (d) AC, and (e) WC.

4. Conclusions

- (1) The lower cooling rate promoted the phase transformation of β to α . Additionally, the phase of the alloy samples transformed from $\beta \rightarrow \alpha$ to $\beta \rightarrow \omega$, and, finally, to $\beta \rightarrow \alpha'$ with increasing cooling rate.
- (2) The average grain size and volume fraction of α phase decreased, and the distribution of α phase varied from compact to scattering with increasing cooling rate. The high cooling rate caused the appearance of α' phase in WC sample and presented a strip structure and crisscross distribution.

- (3) The cooling rate significantly affected the content of Hf in α and β phase. Hf tended to distribute in β phase, and the lower cooling rate was beneficial to the existence of Hf.
- (4) The lower cooling rate improved the plasticity but devastated the strength and microhardness. The elongation first increased, then decreased with increasing cooling rate. Nevertheless, the strength and microhardness enhanced monotonously.

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