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1 **Tensile creep behavior of HfNbTaTiZr refractory high entropy alloy**
2 **at elevated temperatures**

3 Che-Jen Liu^{1,2}, Christian Gadelmeier³, Shao-Lun Lu^{4,5}, Jien-Wei Yeh^{1,6}, Hung-Wei
4 Yen^{4,5*}, Stéphane Gorsse^{7*}, Uwe Glatzel^{3*}, An-Chou Yeh^{1,2,6*}

5 ¹ High Entropy Materials Center, National Tsing Hua University, 101, Sec. 2,
6 Kuang-Fu Road, Hsinchu 30013, Taiwan

7 ² Ph.D. Program in Prospective Functional Materials Industry, National Tsing Hua
8 University, 101, Sec. 2, Kuang-Fu Road, Hsinchu 30013, Taiwan

9 ³ Metals and Alloys, University of Bayreuth, Prof.-Rüdiger-Bormann-Str. 1, 95447,
10 Bayreuth, Germany

11 ⁴ Department of Materials Science and Engineering, National Taiwan University, No.
12 1, Sec. 4, Roosevelt Rd., Taipei 10617, Taiwan

13 ⁵ Advanced Application Centre for Microscopy & Microanalysis, National Taiwan
14 University, No. 1, Sec. 4, Roosevelt Rd., Taipei 10617, Taiwan

15 ⁶ Department of Materials Science and Engineering, National Tsing Hua University,
16 101, Sec. 2, Kuang-Fu Road, Hsinchu 30013 Taiwan.

17 ⁷ Univ. Bordeaux, CNRS, Bordeaux INP, ICMCB, UMR 5026, F-33600 Pessac,
18 France *Corresponding Authors: yehac@mx.nthu.edu.tw;

19 Uwe.Glatzel@uni-bayreuth.de; stephane.gorsse@icmcb.cnrs.fr;

20 homeryen@ntu.edu.tw

21 **Abstract**

22 Tensile creep, which is one of the most important deformation modes for high
23 temperature applications, is rarely reported for refractory high entropy alloys
24 (RHEAs). In the present study, the optical floating zone (OFZ) technique was used to
25 fabricate HfNbTaTiZr with grain size larger than 1 mm on average; tensile creep tests
26 under vacuum at 1100-1250°C and stepwise loading of 5-30 MPa were conducted. The
27 stress exponents and creep activation energies were determined to be 2.5-2.8 and 273
28 $\pm 15 \text{ kJ mol}^{-1}$, respectively. The stress exponents determined have suggested solute
29 drag creep behavior, and deformation was governed by $a/2\langle 111 \rangle$ type dislocations. To
30 elucidate the effect of alloying constituents on solute drag creep, intrinsic diffusion
31 coefficients of all elements were determined by simulation, and theoretical minimum
32 creep strain rates were compared with those of experimental values. Analysis suggests
33 that creep rate of HfNbTaTiZr appears to be controlled by Ta, which possesses the
34 lowest intrinsic diffusivity and contributes the most to drag dislocations. To our
35 knowledge, this work is the first to report tensile creep deformation mechanism of
36 HfNbTaTiZr, especially up to 1250°C.

37

38 Keywords: Refractory high entropy alloy, Creep, High-temperature deformation,

39 Diffusion, Thermally activated processes

40 1. Introduction

41 In the early 2000s, the notion of “high entropy alloys” (HEAs) was
42 conceptualized by Yeh et al. [1] and Cantor et al. [2], and it emerged as a new strategy
43 of alloy design. The composition space of alloys that can be explored is literally
44 unlimited with this approach [3-5]. The microstructures of HEAs can also be
45 customized to meet desired properties, including single phase solid solution HEAs [3,
46 4, 6-8] and HEAs with hierarchical microstructures [9-12]. Furthermore, second
47 phases can be introduced in systems, such as Co-Cr-Fe-Ni-Ti [13, 14],
48 Al-Co-Cr-Fe-Ni [15-22], and high entropy superalloys [23-25]. To propose a potential
49 solution with temperature capability beyond-superalloys, Senkov et al. [26, 27]
50 reported refractory high entropy alloys (RHEAs) that constitute mainly refractory
51 elements, and have since drawn lots of interests to study their mechanical properties,
52 however, reported literatures were mainly based on studies of compression
53 deformation [28-34]. Among all RHEAs, HfNbTaTiZr has attracted lots of attentions,
54 because it possesses a fully disordered BCC solid solution structure with melting
55 point around 1800°C [29, 35]. HfNbTaTiZr exhibits a combination of high strength
56 and ductility at room temperature [29, 30], and at cryogenic temperatures [36].
57 HfNbTaTiZr can also retain its compression strength at elevated temperatures, i.e.
58 790 MPa at 400°C and 675 MPa at 600°C [30]; it has been reported that $a/2\langle 111 \rangle$

59 screw dislocations movements dominate its compression deformation from room
60 temperature to 800°C [37, 38].

61 Tensile creep is one of the most important demands for high temperature
62 applications. However, to the best of authors' knowledge, there has been no literature
63 reported for RHEAs in this subject to-date. Previous works have analyzed the creep
64 behavior of some other refractory alloys. Lin et al. [39] reported creep of Ta-2.5 wt.%
65 W (grain size ~ 70 μm) under 1250–1450°C and a stress range of 35 to 210 MPa. It
66 showed a transition from solute drag creep (stress exponent, $n=3$) to dislocation climb
67 creep ($n>3$) at high-stress regions. Kellner et al. [40] investigated the creep of
68 Mo-9Si-8B based alloys under vacuum at 1250°C with stresses from 50 to 250 MPa;
69 the stress exponent was found to be 3.8, indicating dislocation climb controlled creep.
70 And, C103, which is a Nb-based alloy was reported to creep by solute drag
71 mechanism under high temperature and low stress conditions [41]. It appears that
72 refractory alloys exhibit various creep mechanisms under different testing conditions.
73 Despite a lack of thorough understanding regarding the creep deformation behavior of
74 RHEAs so far, experiments of HEAs have been conducted to characterize their creep
75 properties [42-50]. However, majority of these studies were based on 3d-transition
76 metals based HEAs, only Kral et al. [46] reported the compression creep behavior of
77 AlTiVNbZr_{0.25} RHEA (B2 matrix + Zr₅Al₃-precipitates) at 800°C and stresses of

78 100-560 MPa; its creep mechanism was elucidated to be solute drag creep by
79 analyzing the stress exponent ($n=3$) and an observation of sluggish subgrains
80 formation. So far, there is no report about tensile creep of RHEAs, since RHEAs is a
81 potential candidate for beyond-superalloys applications, it is important to understand
82 the mechanism of tensile creep deformation of the intrinsic high entropy single phase
83 solid solution. In this work, HfNbTaTiZr has been chosen for tensile creep studies at
84 temperatures up to 1250°C, because of its stability as a single BCC solid solution
85 phase at elevated temperatures, unlike the other RHEAs that exhibit complex phases
86 [33, 34, 46, 51] or inversion between B2 and BCC phase [32, 52].

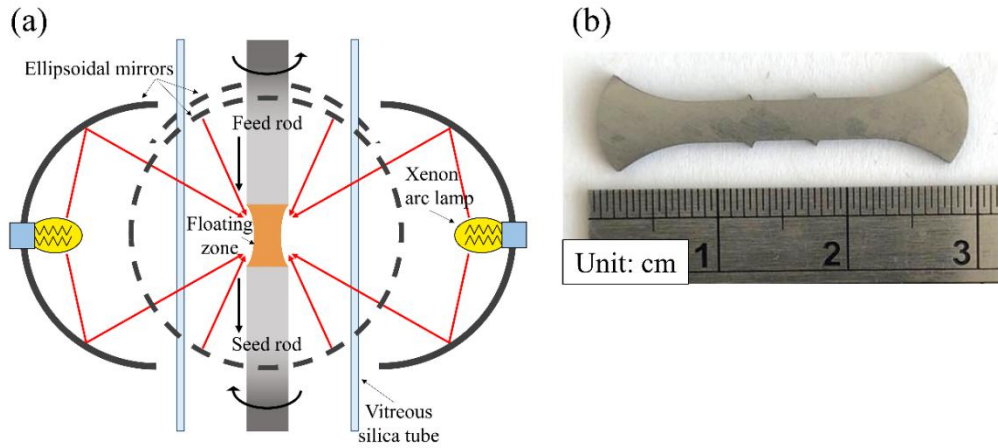
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88 **2. Experimental procedure**

89 The HfNbTaTiZr refractory high entropy alloy was prepared from pure elements
90 Hf, Nb, Ta, Ti, Zr with 99.95 wt.% purity. Ingots were produced by vacuum
91 arc-melting on a copper water-cooled cavity. Before alloying, the arc-melting tank
92 was evacuated to 2 Pa, then filled with argon with 99.99% purity, and evacuated again;
93 this process was repeated three times in order to purge the residual atmosphere, and a
94 pure argon atmosphere filled to 34.5 kPa was achieved. Pure Ti ingot was then
95 re-melted inside the chamber to act as getter of residual oxygen and nitrogen. During
96 the alloying and melting process, each ingot was flipped and melted five times to

97 improve the chemical homogeneity, and ingots with dimensions of $15 \times 35 \times 120 \text{ mm}^3$
98 were produced. Cylindrical rods with 12 mm in diameter and 100 mm in length were
99 obtained from ingots by electrical discharge machining (EDM). Surfaces of the
100 cylindrical rods were then grinded by SiC sandpapers to 800 grit and ultrasonic
101 cleaned in alcohol to ensure no oxide layer remained. The cylindrical samples of
102 HfNbTaTiZr were subjected to crystal growth process by using an optical floating
103 zone (OFZ) furnace (model FZ-T-12000-X-VII-VPO-PC, Crystal Systems
104 Corporation, Japan). OFZ technique was chosen in this work in order to fabricate
105 coarse grains with homogeneous composition [53], and samples with large grains
106 were used to minimize the effect of grain boundary softening in HfNbTaTiZr reported
107 in previous works [30, 54, 55]. The schematic drawing of the four mirror OFZ furnace
108 used in this work is presented in Fig. 1(a). High-purity argon (99.995%) was filled
109 inside the chamber at 101.3 kPa with 3 liters/min flux throughout the process. The
110 pulling rate of the crystal was 15 mm/h, and the rotation rates was 3 rpm to ensure the
111 temperature homogeneity during crystal growth.

112



113

114 Fig. 1. (a) The schematic drawing of the optical floating zone furnace. (b) The tensile
 115 creep test specimen.

116

117 The tensile creep specimens were manufactured by EDM, and they had a
 118 dog-bone shape with the gage-section dimensions of $5 \times 2.9 \times 1.5 \text{ mm}^3$ and four
 119 ridges marking the gage-section, Fig. 1(b). The flat sides of gage were grinded by
 120 1200 grit SiC sandpaper. The vacuum tensile creep test device was hand-built by
 121 Metals and Alloys, University of Bayreuth, details of the setup was described by
 122 earlier reports [40, 56]. The device contains a graphite-heating element inside the
 123 vacuum chamber and a type-S thermocouple close to the specimen. The tensile creep
 124 tests were conducted at 1100, 1150, 1200, and 1250°C in vacuum of $2 \times 10^{-4} \text{ Pa}$
 125 controlled by an oil diffusion pump. The diffusion pump ran over the whole duration
 126 of creep tests to guarantee a high vacuum level, which could prevent reaction with C,
 127 O_2 , or N_2 and minimize contamination during high temperature creep tests. Two Al_2O_3

128 ceramic holders were used to grip the ends of the testpieces. A non-contacting video
129 extensometer was used to record the strain evolution during creep [56]. In this work,
130 creep tests were conducted with step-wise increase in stress; the change in load was
131 determined by correlating the instantaneous cross-sectional area after the minimum
132 creep rate was reached, this method was described in details by Gadelmeier et al. [49].
133 The tensile creep tests were conducted under stresses of 5, 10, 20, and 30 MPa, Table
134 1. The samples were first tested at 1100°C and 10 MPa; after the strain rate had
135 reached the minimum creep rate, the stress was raised to 20 MPa, and then from 20
136 MPa to 30 MPa. The other stress change tests were also conducted with the load
137 increased stepwise from 5, 10 to 20 MPa at 1150°C, 1200°C, and 1250°C. The load
138 change process took less than 2 minutes. After tests, all creep specimens were cooled
139 freely to room temperature by furnace cooling. During the cooling process, from
140 testing temperatures to 940°C, the cooling rates were about 260°C/min; from 940°C to
141 800°C, and 800°C to 600°C, cooling rates were 85°C/min and 32°C/min, respectively,
142 below 600°C, the cooling was around 5-12°C/min.
143

144 Table 1. Testing temperatures and corresponding stresses of tensile creep tests.

Temperature (°C)	Stress (MPa)
1100	10, 20, 30
1150	5, 10, 20
1200	5, 10, 20
1250	5, 10, 20

145

146 To reveal the microstructures, specimen surfaces were grinded by SiC sandpaper
 147 to 4000 grit, followed by 0.05 μm Al_2O_3 and 0.02 μm silica suspension polishing steps.
 148 Etching was conducted with a hydrofluoric acid solution (20 ml hydrofluoric acid +
 149 15 ml nitric acid + 65 ml glycerol). For grain size analysis, an optical microscope was
 150 used, and an average grain size was determined by ImageJ 1.42q [57]. X-ray
 151 diffraction (XRD, D2 PHASER X-ray diffractometer, Bruker) with Cu $K\alpha$ radiation
 152 was used to conduct phase analysis. Scanning electron microscopes (SEM,
 153 JSM-7610F, JEOL and Gemini 300, Zeiss) equipped with electron backscatter
 154 diffraction (EBSD) and energy-dispersive X-ray spectroscopy (EDS) were used to
 155 examine the microstructures in details. The backscatter Kikuchi patterns were
 156 recorded by AZtec EDS/EBSD software (Oxford Instruments plc, UK) to map the
 157 misorientaion profiles, and kernel average misorientaion (KAM) mapping was

158 conducted by calculating the arithmetic average of the scalar misorientation between
159 groups of pixels [58]. Dual-beam focused ion beam (FIB, Helios Nanolab 600i, FEI,
160 Oregon, USA) was used to prepare samples for transmission electron microscope
161 analysis (TEM, FEI TECNAI G2 F20 TEM) operating at 200 kV. To elucidate the
162 effects of alloying constituents in HfNbTaTiZr on creep rates, thermodynamic
163 database TCHEA4 and mobility database MOBHEA2 were used to estimate the
164 intrinsic diffusivities of each element in the system, so the theoretical creep rates
165 could be determined to compare with those of experimental values for discussion.

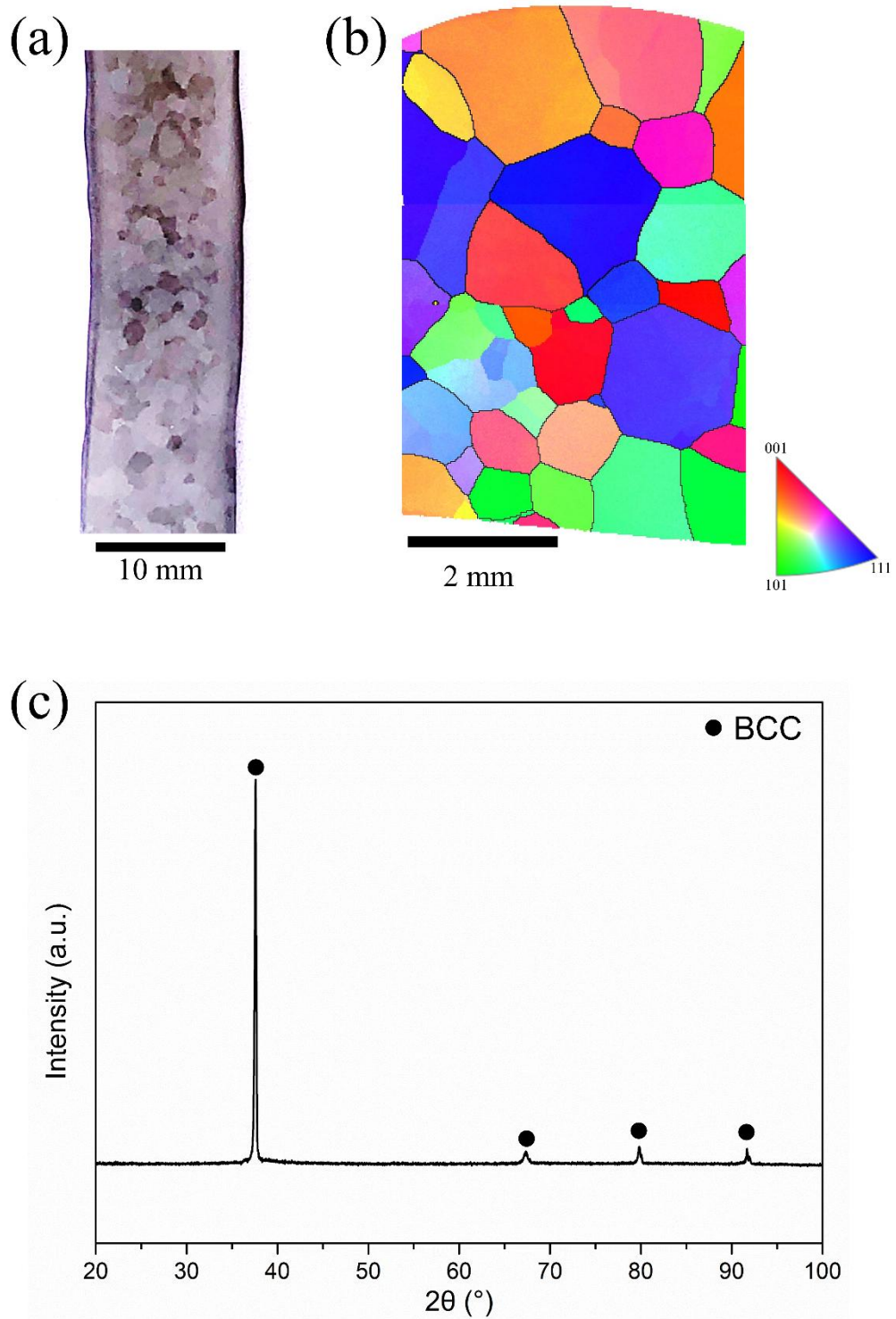
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167 **3. Results and analysis**

168 3.1 Microstructure prior creep

169 The sample of HfNbTaTiZr processed by OFZ furnace had an average grain size
170 of 1.2 ± 0.4 mm, Fig. 2(a) and 2(b), and this was substantially coarser than the grain
171 sizes of this RHEA reported in previous works fabricated by arc-melting (140 μm)
172 [54], spark plasma sintering (20-190 μm) [59, 60], and hot isostatic pressing (100-200
173 μm) [29, 30]. The microstructure of the sample exhibited a BCC single phase with a
174 lattice parameter of 340 pm, Fig. 2(c). There was no dendritic segregation observed
175 and the equi-atomic compositions of HfNbTaTiZr was confirmed by SEM-EDS.

176



177

178 Fig. 2. (a) Longitudinal section and (b) Inverse pole figure (IPF) image of the cross

179 section of HfNbTaTiZr. (c) X-ray diffraction pattern of sample prior creep.

180

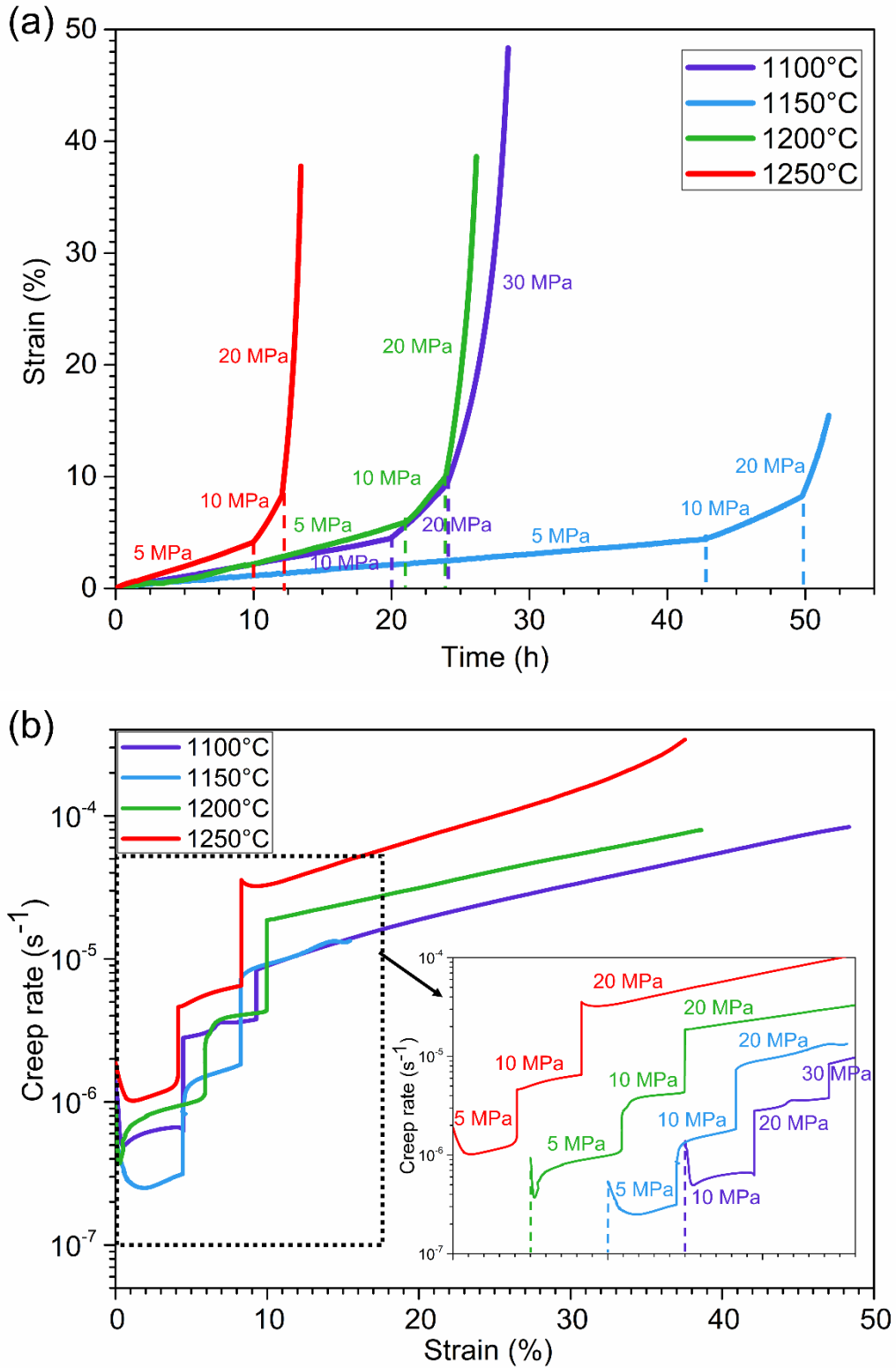
181 3.2 Creep behavior

182 Creep curves are shown in Fig. 3(a), and the evolutions of creep rates versus creep strain are shown in Fig. 3(b). The purpose of this study was to minimize the grain boundary softening, so the intrinsic creep mechanism of HfNbTaTiZr could be elucidated. Although the texture of coarse grains has been known to influence creep strains and creep lives [61], this study focused on elucidating creep mechanism based on the minimum creep strain rates. The minimum creep rates have been determined and summarized in Table 2. Creep tests under each condition were performed twice to ensure reproducibility, the minimum creep rates of two sets of tests were almost the same under the same stress and temperature. So, this indicates that the use of coarse grain structure of HfNbTaTiZr in this work could result consistent minimum creep strain rates under present testing conditions.

192 The minimum creep rate can be used to determine the stress exponent n and the creep activation energy Q .

$$\dot{\epsilon}_{min} = A\sigma^n \exp\left(-\frac{Q}{RT}\right) \quad (3-1)$$

193 where $\dot{\epsilon}_{min}$ is the minimum creep rate, A is a material specific constant, σ is the applied stress, R is the gas constant, and T is the absolute temperature.



195

196 Fig. 3. (a) Tensile creep curves, (b) Creep rate versus strain.

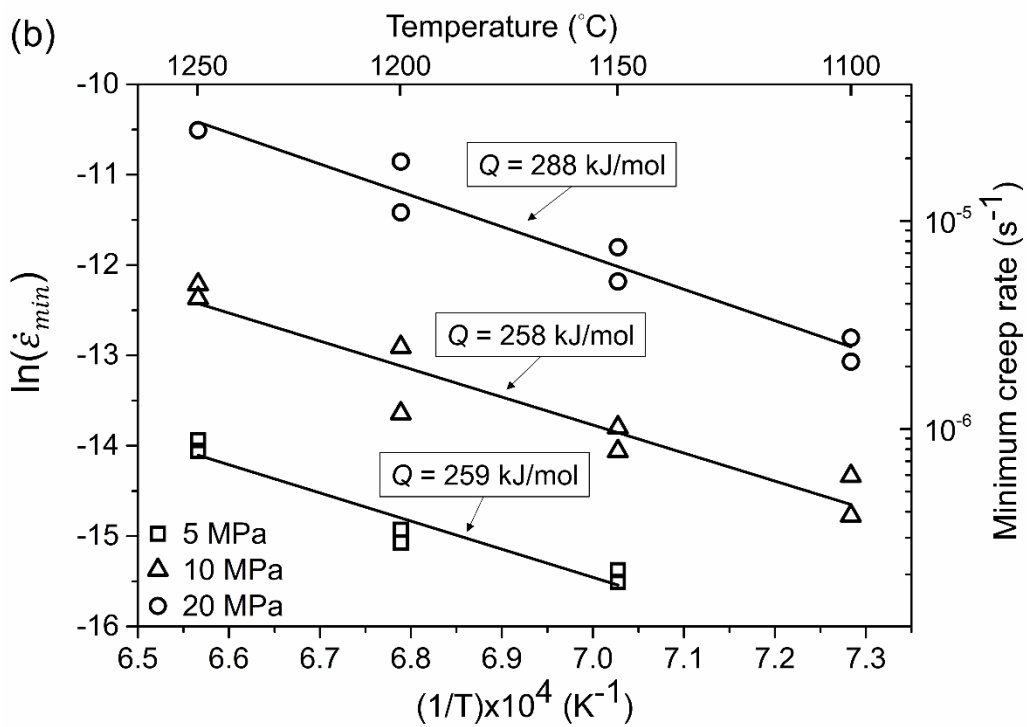
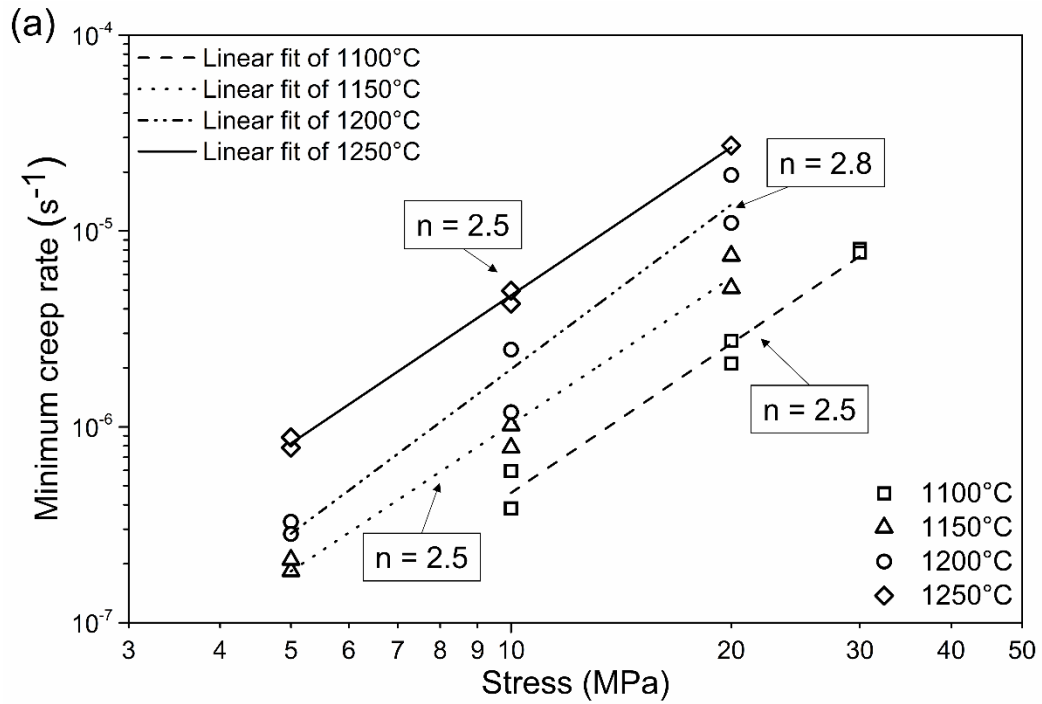
197

198 Table 2. The minimum creep rates of HfNbTaTiZr at different loads and temperatures.

Log₁₀ minimum creep rate (s⁻¹)								
T (°C)	1100		1150		1200		1250	
Load (MPa)	Test 1	Test 2	Test 1	Test 2	Test 1	Test 2	Test 1	Test 2
5	-	-	-6.7	-6.7	-6.5	-6.5	-6.1	-6.1
10	-6.4	-6.2	-6.1	-6.0	-5.6	-5.9	-5.4	-5.3
20	-5.7	-5.6	-5.3	-5.1	-4.7	-5.0	-4.6	-
30	-5.1	-5.1	-	-	-	-	-	-

199

200 By plotting the minimum creep rates against the applied stresses, Fig. 4(a),
 201 values of the stress exponent n can be determined from slopes of linear regression
 202 lines. The activation energy Q for creep can be determined by plotting the natural log
 203 of the minimum creep rate at a specific stress versus the reciprocal of temperature, Fig.
 204 4(b). The values of n and Q and their standard deviations are summarized in Table 3.
 205 The stress exponent values in this work vary in the range of 2.5 to 2.8, which suggests
 206 the solute drag creep mechanism [63, 64], and the activation energy is determined to
 207 be 273 ± 15 kJ mol⁻¹. There was no transition between different creep mechanisms
 208 within the stresses and temperatures regime in this investigation.



209

210 Fig. 4. (a) Minimum creep rates versus applied stress from 1100 to 1250°C, (b)

211 minimum creep rates versus the reciprocal of the absolute temperatures from 5 to 20

212 MPa.

213 Table 3. Calculated power-law parameters, n , Q and coefficients of determination R^2 .

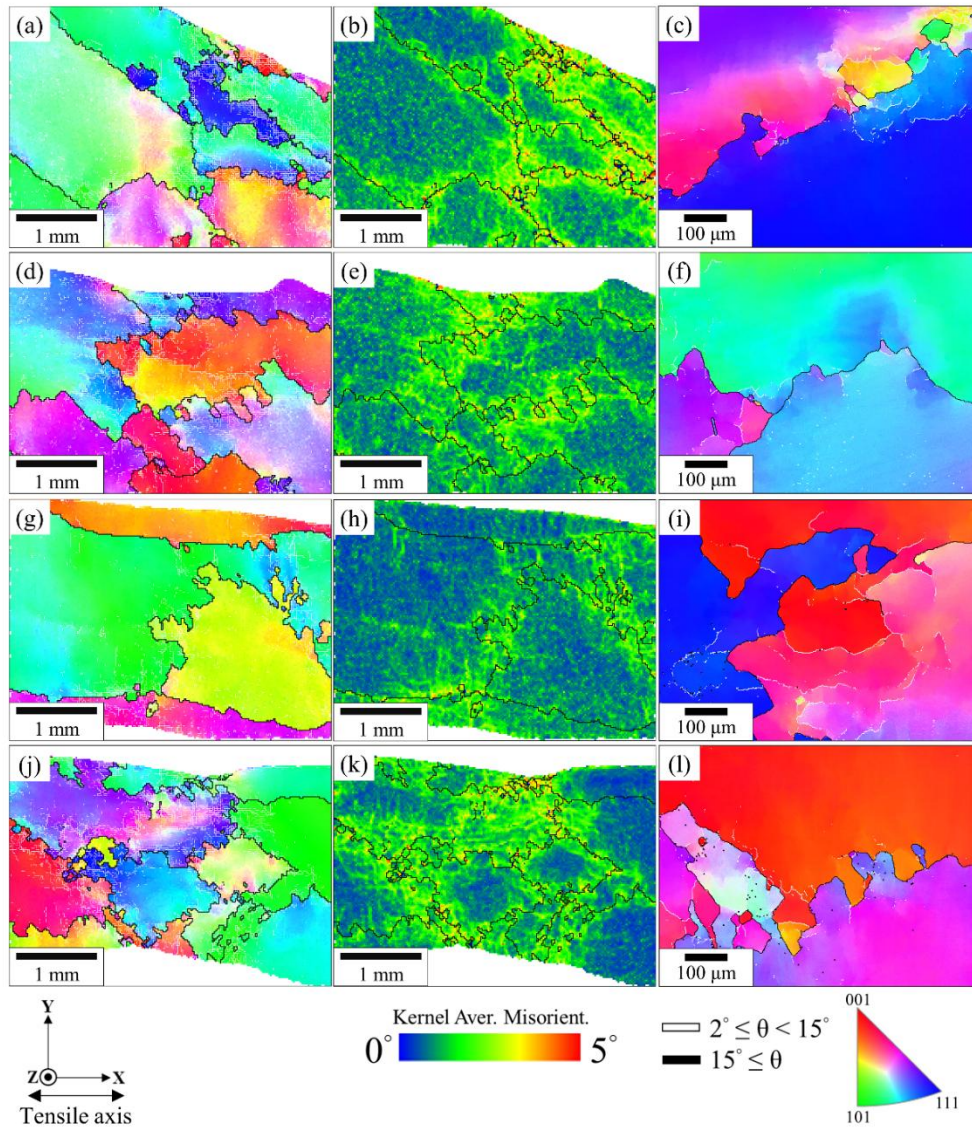
T (°C)	n	R^2
1100	2.5	0.9747
1150	2.5	0.9820
1200	2.8	0.9600
1250	2.5	0.9968
Stress (MPa)	Q (kJ mol⁻¹)	R^2
5	259	0.9147
10	258	0.8979
20	288	0.9380

214

215 3.3 Microstructures after creep

216 The microstructure of crept HfNbTaTiZr evolved significantly after deformation,
 217 Fig. 5; grain morphology was distinctly different from the microstructure prior creep
 218 shown in Fig. 2. Fig. 5 shows that significant bulging of grain boundaries occurred
 219 during creep, and these bulging grain boundaries were associated with high levels of
 220 misorientation, which indicates concentration of deformation in the vicinity of grain
 221 boundaries. This type of deformed grain boundaries in HfNbTaTiZr was reported
 222 previously during the early stage of uniaxial compression testing at 1000°C [55], and
 223 continuing deformation under compression to over 30% would lead to dynamic
 224 recrystallization in necklace form along initial grain boundaries [30, 54, 55]. However,

225 the necklace structure at boundaries was not identified after creep in this study, Fig. 5.
226 Furthermore, subgrain boundaries were observed near bulging grain boundaries due to
227 polygonization, Fig. 5 (c, f, i, l), this phenomena was observed in other metals
228 deformed in the solute drag regime [65, 66]. Despite significant deformation of grain
229 boundaries after creep, there was no crack or void observed at the intergranular
230 regions.
231



232

233 Fig. 5. SEM-EBSD images of the specimens after creep under different temperatures:

234 (a, b, c) 1100°C, (d, e, f) 1150°C, (g, h, i) 1200°C and (j, k, l) 1250°C; (a, d, g, j) are

235 inverse pole figure maps, (b, e, h, k) are kernel average misorientation maps and (c, f,

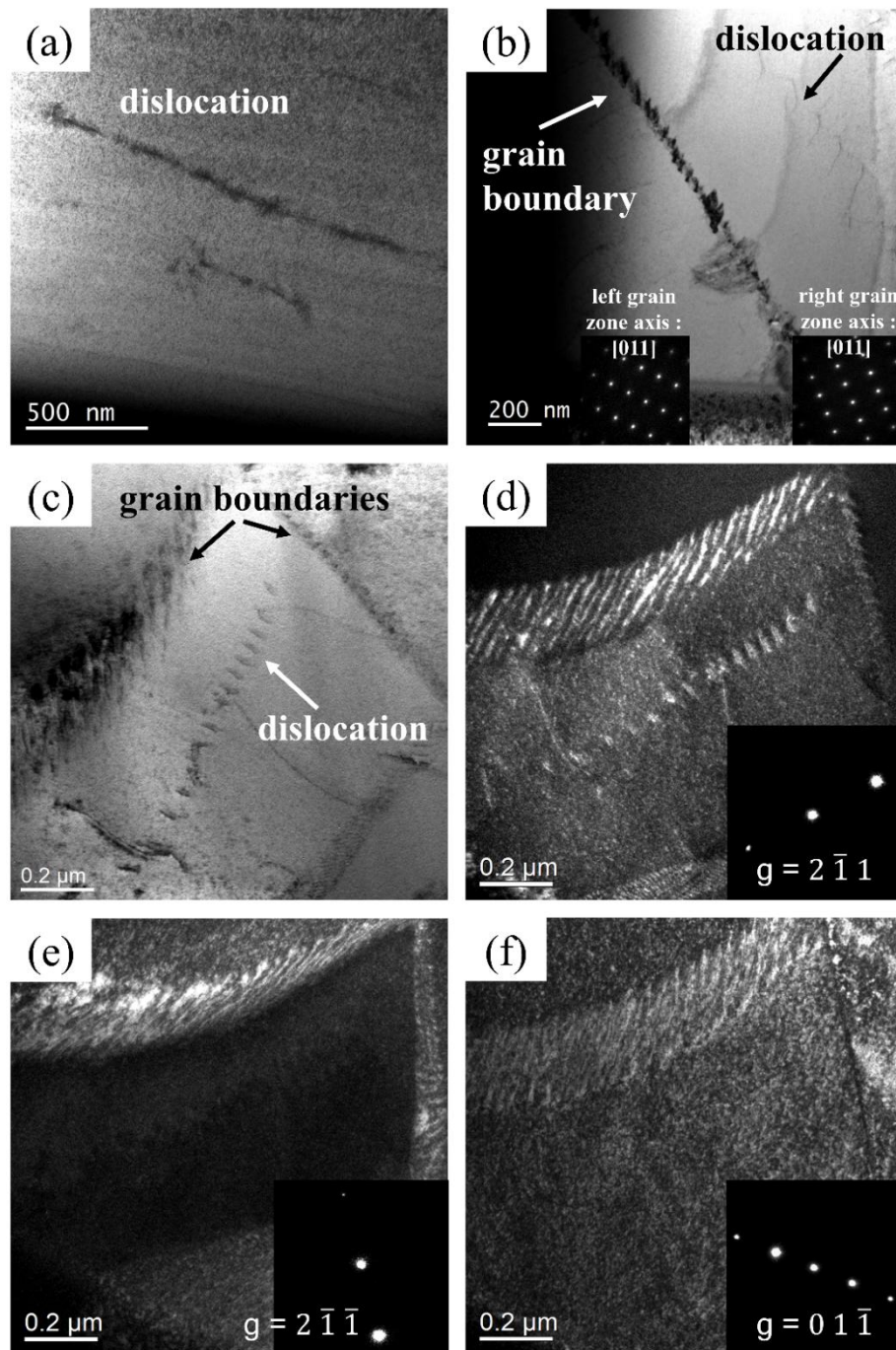
236 i, l) are inverse pole figure maps under higher magnification.

237

238 To further examine the crept microstructures, TEM images were analyzed. Since

239 all creep conditions from 1100 to 1250°C shared similar deformation behavior and

240 stress exponent values, the dislocation structures of HfNbTaTiZr after creep at
241 1100°C/30 MPa with 46% strain are shown exemplarily in this work, Fig. 6. The
242 dislocations were randomly distributed, and they were long and neatly arranged
243 without entanglement. There were also dislocations accumulation along the grain
244 boundaries, Fig. 6(b), the selected area diffraction patterns confirmed that deformed
245 HfNbTaTiZr still retained its BCC structure, exemplified by the [011] zone axis. In
246 addition, the Burger's vector (b) of the gliding dislocations was determined by TEM
247 analysis with the $g \cdot b$ invisibility criterion. The dislocations were visible with
248 $\vec{g} = [2\bar{1}1]$ in Fig. 6(d), while they were out of contrast with $\vec{g} = [2\bar{1}\bar{1}]$ in Fig. 6(e)
249 and $\vec{g} = [01\bar{1}]$ in Fig. 6(f). This analysis has validated that dislocations in tensile
250 creep deformed HfNbTaTiZr were $a/2\langle 111 \rangle$ type.



251

252 Fig. 6. TEM images of HfNbTaTiZr after creep at 1100°C. (a) dislocation structure

253 within grain interior; (b, c) overview of dislocation and grain boundary structure after

254 creep; (d, e, f) Weak-beam dark-field micrographs from the same area of (c) in zone

255 axis near (d) $[1\bar{1}\bar{3}]$, (e) $[13\bar{1}]$ and (f) $[1\bar{1}\bar{1}]$.

256 4. Discussion

257 The stress exponent n can be used to elucidate the dominant creep mechanism.
258 For HfNbTaTiZr tested at 1100-1250 °C and 5-30 MPa, the stress exponents were
259 in-between 2.5-2.8, which is slightly lower than 3. The stress exponent about 3 is
260 known to be related to solute drag creep (viscous glide creep or the Class I solid
261 solution creep behavior), and solute drag creep is often observed for solid solution
262 alloys [63, 64]. The solute drag creep effect can additionally be raised from
263 dislocation interactions with solute atoms and retard dislocation glide movement [62,
264 64, 67-69]. Furthermore, a transition from solute drag creep to climb-controlled creep
265 would be observed with the breakaway of dislocations from the stress field of solute
266 with higher stresses [63]. Since the stress exponents determined from all creep
267 conditions were similar, the creep mechanism did not change within different
268 temperatures and stresses in this study. This indicates that the stress level in this work
269 was not high enough to overcome the solute drag effect.

270 In addition to the stress exponent, the microstructures after creep in Fig. 6 also
271 suggest solute drag creep in present study. Since the completion of creep tests were
272 followed by sufficient cooling rate (~260°C/min from testing temperatures to 940°C),
273 the dislocation structures would be retained after creep tests. The observed
274 dislocations were slightly curved without entanglement; this observation is the

275 typically identified in samples deformed by solute drag creep mechanism [46, 63, 65].

276 Bulging grain boundaries and vast levels of misorientation along grain
277 boundaries after creep, Fig. 5, have been shown by KAM analysis. Based on the
278 deduced creep mechanism, the grains were deformed by dislocations, Fig. 6,
279 dislocations piling up at the grain boundaries were also observed, Fig. 6(b) and 6(c).
280 The variation of dislocation densities at grain boundaries activated the bulging of
281 grain boundaries at different segments, which is known as the strain induced grain
282 boundary migration [55, 70]. The evolution of microstructure of HfNbTaTiZr after
283 deformation in this work is different from those reported previously [30, 54, 55]. In
284 the work conducted by Senkov et al. [30], dynamic recrystallization was observed
285 after deformation; HfNbTaTiZr with initial grain size of 100-200 μm was subjected to
286 50% height reduction under compression at temperatures up to 1200°C, the strain rate
287 was 10^{-3} s^{-1} and the stress could reach 92 MPa and above. Eleti et al. [54] also
288 reported dynamic recrystallization of 50% height reduction hot-compressed
289 HfNbTaTiZr with an average initial grain size of 140 μm , the strain rate was 10^{-4} s^{-1}
290 and the stress could reach 44 MPa and above. The evolution of microstructure under
291 compression was further clarified in another work by Eleti et al. [55]; with the strain
292 rate of 10^{-3} s^{-1} , bulging grain boundaries associated with strain induced grain
293 boundary migration were observed during the early stage of compression deformation

294 under 10% strain at 1000°C, and there was no dynamic recrystallization reported until
295 30% reduction. The dynamic recrystallization in those compression tests could evolve
296 from around the initial grain boundaries in necklace form to fully recrystallization
297 with reduction ratios greater than 30%. By contrast, the mode of deformation in this
298 work was creep in tension under relatively lower stresses (5-30 MPa), with creep
299 strains of 15 to 48 % and above, the stress build-up at the grain boundaries should be
300 lower than those subjected to compression deformation [30, 54, 55], hence bulging
301 grain boundaries were observed with no dynamic recrystallization, Fig. 5.
302 Furthermore, the grain size in this work (1.2 ± 0.4 mm) was about 10 times larger
303 than those of Senkov et al. [30] and Eleti et al. [54], so deformation attributed to
304 diffusion along grain boundary can be less. Hence the tensile creep activation energy
305 (273 ± 15 kJ mol⁻¹) determined in the present work is higher than the activation
306 energies of compression deformation reported by Senkov et al. [30] (226 ± 20 kJ
307 mol⁻¹) and Eleti et al. [54] (245 ± 13 kJ mol⁻¹).

308 All our observations highlight that the tensile creep mechanism of HfNbTaTiZr
309 at elevated temperatures is glide-controlled creep by solute drag. This can also refer to
310 the case in which dislocations encounter frictional stress acting on it by dragging the
311 solute stress fields [63, 64, 71]. According to Mohamed et al. [64], the minimum
312 creep rate can be modeled using the following equation:

$$\dot{\epsilon}_{min} \quad (4-1)$$

313 where ν is the Poisson's ratio, σ is the applied stress, G is shear modulus of the
314 alloy, and A represents the interaction of dislocations with solute atoms which is
315 expressed as Equation (4-2) according to Cottrell et al. [71]:

$$A \quad (4-2)$$

316 where e is the solute-solvent size difference, c is the concentration of solute atoms, b
317 is Burger's vector, k is Boltzmann's constant, T is the absolute temperature, and \tilde{D}
318 is the diffusion coefficient of solute atoms. Combining Equations. (4-1) and (4-2) can
319 give the following expression to predict the minimum creep rate [63]:

$$\dot{\epsilon}_{min} \quad (4-3)$$

320 According to solute drag by the Cottrell-Jaswon mechanism (Equation (4-3)), the
321 dislocation could drift under the action of stress acting on it by dragging behind it a
322 solute atmosphere [71]. The solute atmosphere could travel by means of the diffusion
323 of the solute atoms. If the dislocation was to move at a given velocity, then the solute
324 atoms must diffuse at the same velocity [63]. Although Equation (4-3) has been
325 commonly used in conventional alloys, Kim et al. [45] adopted this equation by
326 considering each constituent as the solute atmosphere and made a direct comparison
327 between the calculated values and the observed minimum creep rates. The results
328 suggested that the solute drag of Ni controlled the viscous glide during creep of

329 CoCrFeMnNi. Therefore, this work has applied the same approach to determine the
330 solute which would dominate the dragging mechanism during creep of HfNbTaTiZr.
331 However, what should be the appropriate diffusion coefficients, \tilde{D} , of HfNbTaTiZr
332 for Equation (4-3)? Even though a similar question was previously discussed in binary
333 systems [64, 72-76], this has remained an issue of debate. In concentrated
334 multicomponent solid solutions, the determination of the appropriate diffusion
335 coefficient can be even more challenging. Heckl et al. [77] tackled the problem in
336 Ni-based superalloys and considered the weighted harmonic mean of the diffusion
337 coefficients of alloying constituents by referring to the treatments from Herring [72]
338 and Reed et al [78]. However, these different authors considered three different
339 diffusion coefficients, i.e., intrinsic, tracer, or chemical diffusion coefficients, which
340 can be questionable in this approach. Besides, the invoked Herring's equation [72] is
341 valid for climb-controlled creep only, not glide. A diffusion coefficient is, by
342 definition, the proportionality factor between the flux and the concentration gradient.
343 In a 5-components system, the flux equations give a 4x5 intrinsic diffusivity matrix
344 (in the lattice-fixed frame of reference) to describe the diffusion process. To simplify
345 this complicated problem, cross terms can be neglected by postulating small
346 concentration gradients between the segregation surrounding the dislocation and the
347 concentrated solid solution matrix. Only five intrinsic (individual) diffusion

348 coefficients are then sufficient, so the observed minimum creep rates with those
 349 inferred from the individual diffusion coefficient of each constitutive element using
 350 Equation (4-3) can be compared and determine possible elemental effects. Table 4
 351 provides the parameters required to calculate the minimum creep rates by Equation
 352 (4-3). The composition and temperature dependencies of diffusivities for individual
 353 elements in HfNbTaTiZr have been computed using the thermodynamic TCHEA4 and
 354 mobility MOBHEA2 database. The calculated diffusion coefficient of Zr in
 355 HfNbTaTiZr at 1150°C in this work is similar to an experimental result reported
 356 recently by Zhang et al. [79].

357

358 Table 4. The values of intrinsic diffusion coefficients (D_i) for individual elements in
 359 HfNbTaTiZr calculated at different temperatures using ThermoCalc and the TCHEA4
 360 and MOBHEA2 databases, atomic radius (r) [80, 81] and solute-solvent size
 361 difference (e).

	Hf	Nb	Ta	Ti	Zr
Intrinsic diffusion coefficients, $\log_{10}(\text{m}^2/\text{s})$					
1100 °C	-12.3	-13.6	-16.0	-13.4	-13.7
1150 °C	-12.0	-13.3	-15.7	-13.1	-13.4
1200 °C	-11.7	-13.0	-15.3	-12.8	-13.1
1250 °C	-11.5	-12.7	-15.0	-12.5	-12.8
Atomic radius					
r (pm)	157.8	142.9	143.0	146.2	160.3
Solute-solvent size difference					
e (%)	+5.2	-4.8	-4.7	-2.6	+6.8

362

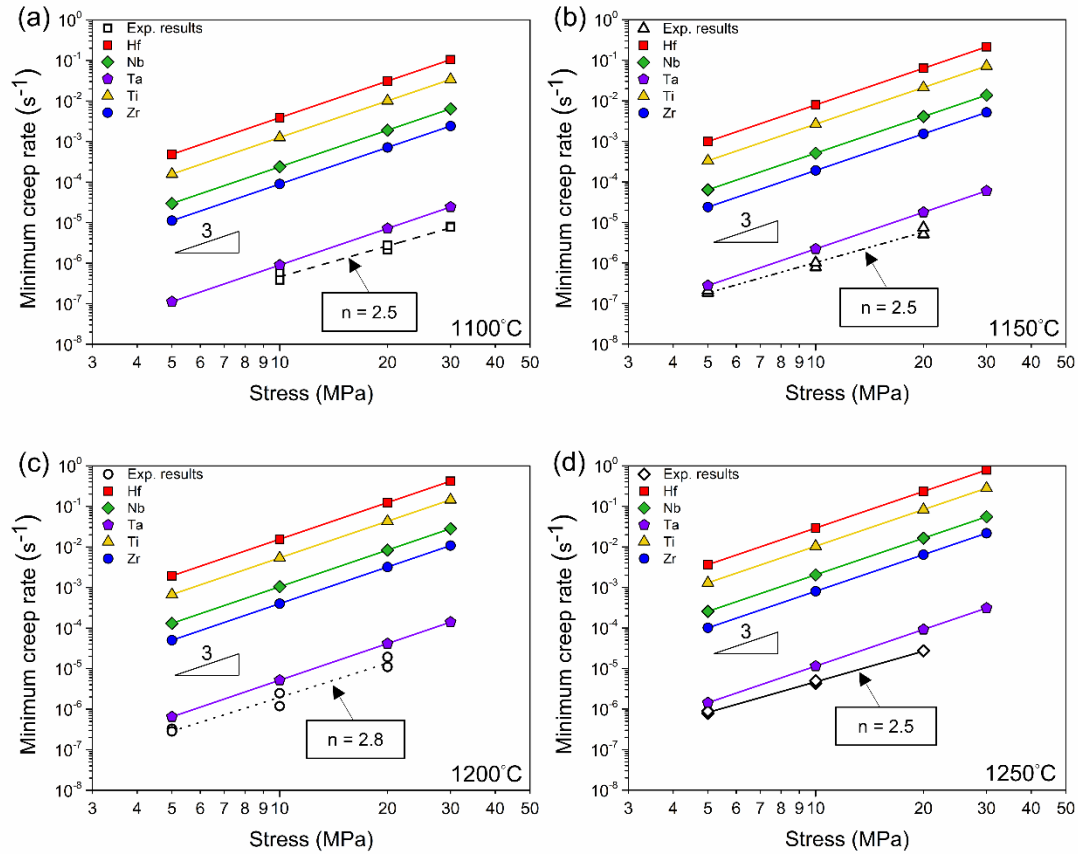
363 The size difference of each element (e_i) can be determined from the following
364 equation [45]:

$$e_i \quad (4-4)$$

365 where r_i is the radius of the element of i , and $r_{average}$ is the average radius of the
366 constituent atoms according to atomic fraction (X_i) of HfNbTaTiZr ($\sum_{i=1}^5 X_i r_i$).

367 The shear modulus of HfNbTaTiZr has been estimated to vary from 30 to 28 GPa
368 between 1100 and 1250°C according to $G = 36.2 - 5.6/(e^{845/T} - 1)$ [82]. The
369 value of 0.26 for the Poisson's ratio can be taken from previous work [82], and the
370 Burger's vector can be calculated from the lattice constant in Fig. 2(c), $b = a/2\langle 111 \rangle$
371 = 0.294 nm. The minimum creep rates can then be calculated at 1100, 1150, 1200 and
372 1250°C by considering each intrinsic diffusion coefficient separately. The results are
373 illustrated in Fig. 7.

374



375

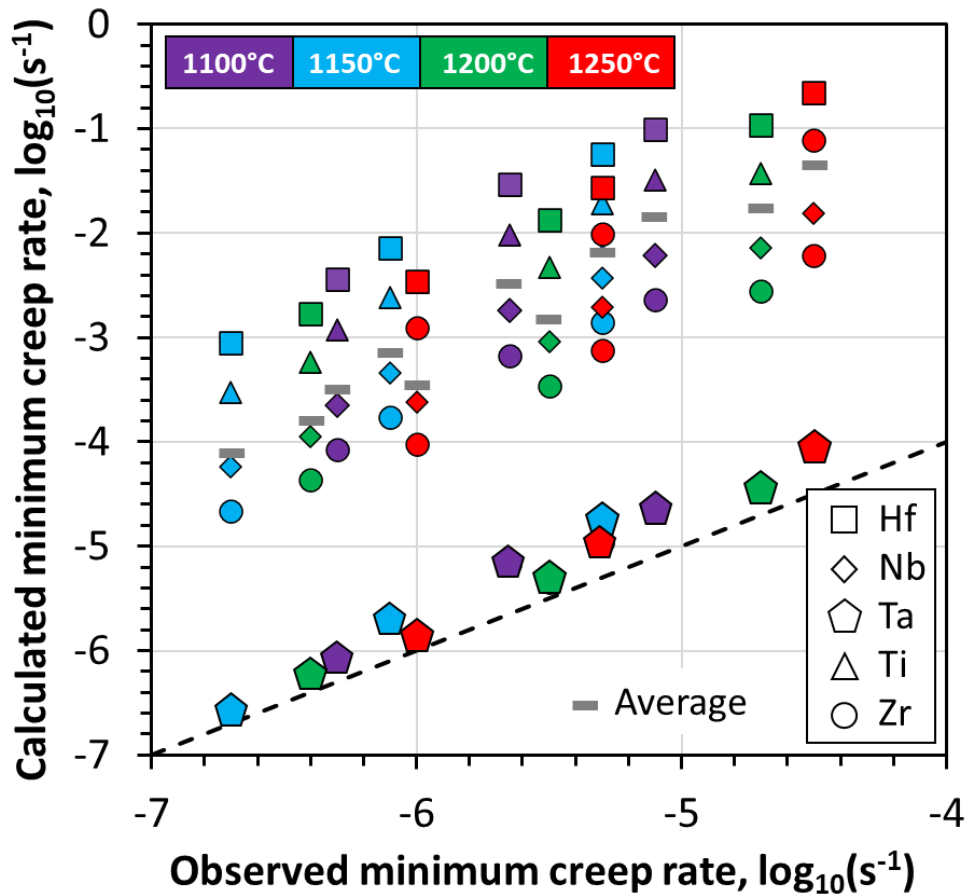
376 Fig. 7. The minimum creep rates against applied stress for the experimental work and
 377 the calculated ones at (a) 1100°C, (b) 1150°C, (c) 1200°C and (d) 1250°C.

378

379 The calculated minimum creep rates in Fig. 7 shows that $\dot{\epsilon}_{min-Ta} < \dot{\epsilon}_{min-Zr} <$
 380 $\dot{\epsilon}_{min-Nb} < \dot{\epsilon}_{min-Ti} < \dot{\epsilon}_{min-Hf}$. Although the experimental results plotted in Fig. 7 are
 381 slightly slower than the $\dot{\epsilon}_{min-Ta}$, it is very similar to the calculated results based on
 382 the intrinsic diffusion of Ta in HfNbTaTiZr, under all stresses and temperatures. Fig. 8
 383 shows that taking the harmonic average of the intrinsic diffusion coefficients in
 384 Equation (4-3) does not agree with the experimentally determined minimum creep
 385 rate. In contrast, the minimum creep rate is predicted with a good agreement with

386 experimental observations using the intrinsic diffusion coefficient of Ta, which
 387 indicates that Ta contributes the most to drag dislocations during creep in
 388 HfNbTaTiZr.

389



390

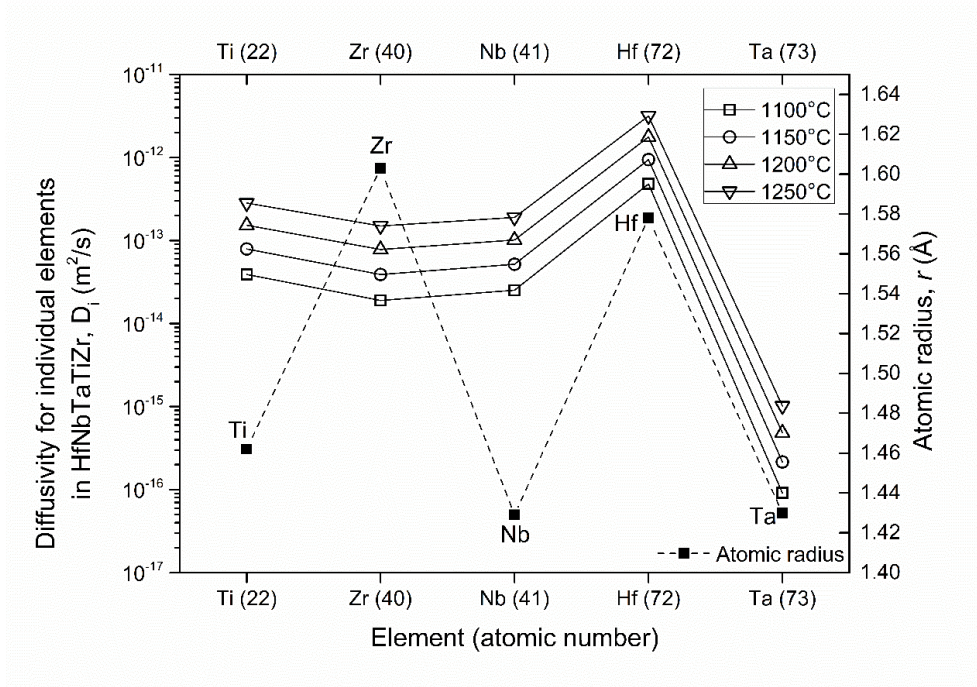
391 Fig. 8. Predicted vs observed minimum creep rates at 1100, 1150, 1200 and 1250°C
 392 and for three different applied stresses between 5 and 30 MPa.

393

394 The diffusion rate in alloy systems is generally believed to be lower when the
 395 solute-solvent size difference of individual elements is larger [83]. However, in Fig. 9,
 396 the diffusivity for individual elements in HfNbTaTiZr from calculation disproves this

397 traditional view. Table 4 shows that Zr and Hf have larger atomic radii, but the
398 diffusivities are faster than Ta in several orders of magnitude. Instead, although Ta
399 does not contribute to the highest size differences, the result implies that the solute
400 drag effect is more dependent on the diffusivity, and Ta has the lowest diffusion
401 coefficients in HfNbTaTiZr and therefore contributes the most to drag dislocations. A
402 similar phenomenon was reported in nickel-base superalloy [84, 85]; Re and Ru have
403 slower diffusion rates in Ni than Zr and Hf, albeit Zr and Hf possess greater atomic
404 radii than Re and Ru, this behavior is thought to be related to the bonding
405 characteristics of solute atoms. Furthermore, the diffusivity is affected by both the
406 thermodynamic factor and the atomic mobility, and low mobility generally comes
407 with a high melting point; the high melting point of Ta may play a role in the observed
408 solute drag creep. Future experimental studies of interdiffusion, solute-vacancy
409 exchange and bonding characteristics in HfNbTaTiZr can help to further understand
410 its high temperature properties. And, single crystals of HfNbTaTiZr would help to
411 further clarify the effect of crystal orientation on deformation behaviors. To our
412 knowledge, this work is the first to unveil the creep deformation mechanism of
413 HfNbTaTiZr in tension, especially up to 1250°C.

414



415

416 Fig. 9. Diffusivity and atomic radius [80, 81] for each element in HfNbTaTiZr RHEA.

417 **5. Conclusion**

418 The current work investigated creep behaviors of HfNbTaTiZr with large grain size
419 fabricated by the optical floating zone technique. Creep properties and microstructure
420 were examined for samples tested at 1100-1250°C and stress levels of 5-30 MPa
421 under vacuum. The important findings are listed as follows.

422 (1) The creep behavior was related to the solute drag mechanism, which is evidenced
423 by the stress exponents of 2.5-2.8. The creep activation energies obtained in this
424 work are in the range of $273 \pm 15 \text{ kJ mol}^{-1}$.

425 (2) TEM characterization has verified that plasticity of creep was dominated by
426 dislocations of $b = a/2\langle 111 \rangle$.

427 (3) Dislocations at grain boundaries have led to significant bulging of grain
428 boundaries associated with strain-induced grain boundary migration.

429 (4) According to the diffusivity calculation and solute drag creep model prediction,
430 the creep rates were controlled by diffusivity of Ta, which is the element with the
431 lowest diffusion coefficient in HfNbTaTiZr and suppresses dislocation movement
432 during creep.

433 These findings can be applied to design advanced RHEAs to improve the creep rate
434 and related rapture.

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