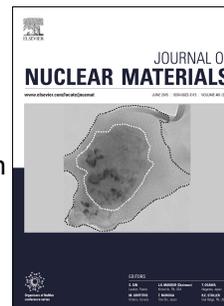


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Texture evolution during annealing of hot extruded U-10wt%Zr alloy by in situ neutron diffraction

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Abstract

Texture evolution during annealing of a U-10wt%Zr alloy hot extruded in the $(\alpha + \delta)$ region was studied by in situ neutron diffraction. The extruded alloy had a lamellar $(\alpha + \delta)$ microstructure and the initial texture consisted of $(100)_\alpha$, $(110)_\alpha$, and the $(0001)_\delta$ poles oriented along the extrusion direction. The β phase, after the $\alpha \rightarrow \beta$ transformation, showed no preferred orientation while the γ phase, after the $\beta \rightarrow \gamma$ transformation, had $(110)_\gamma$ and $(111)_\gamma$ poles oriented along the extrusion direction. After a temperature cycle through the γ phase, there was a complete loss of texture in the α phase while the δ phase retained the initial texture indicating a memory effect.

Keywords: Uranium, Zirconium, Extrusion, Texture, Neutron diffraction

1 Introduction

High thermal conductivity and the ability to incorporate minor actinides makes metallic fuels an attractive choice for fast reactors [1]. Additionally, irradiation data from one of the alloy compositions, i.e., uranium-10wt% zirconium (U-10Zr) alloys available from the

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25 Experimental Breeder Reactor-II (EBR)¹ indicate their viability as a potential fuel form
 26 [2]. U-Zr alloy fuel rods can be produced by different processes like injection casting, ma-
 27 chining, and extrusion. Injection casting requires very high temperatures and expensive
 28 crucibles while machining results in large material losses. Extrusion may be a favourable
 29 alternative to other methods since it is more cost effective. It is a common fabrication
 30 process that is performed on an industrial scale around the world to cost-effectively man-
 31 ufacture many simple shapes from many materials such as aluminum, copper, and steel
 32 [3]. Extrusion of uranium or its alloys is not a common practice; however, it has been
 33 previously investigated to some detail where extruded fuel pins were tested in EBR-I [1].
 34 The present study is aimed towards the evaluation of extrusion as a potential manufac-
 35 turing process for U-10Zr fuel pins by understanding the microstructural changes of a
 36 hot extruded billet during annealing.

37 Uranium and its alloys undergo several phase transformations with temperature: pure
 38 uranium occurs in the orthorhombic α -phase at room temperature, followed by a transi-
 39 tion at 669 °C to the tetragonal β -phase, and at 776 °C to the body centered cubic (bcc)
 40 γ -phase [4]. In the U-Zr system studied here, α , β , γ along with δ are relevant within
 41 the context of this work. Here, the δ -phase is hexagonal and exists along with α at room
 42 temperature (U-Zr phase diagram is shown in Fig. 1). Because of the strong thermo-
 43 elastic and plastic anisotropy exhibited by α -U, any thermo-mechanical processing in the
 44 α phase results in the texture development [5, 6, 7].

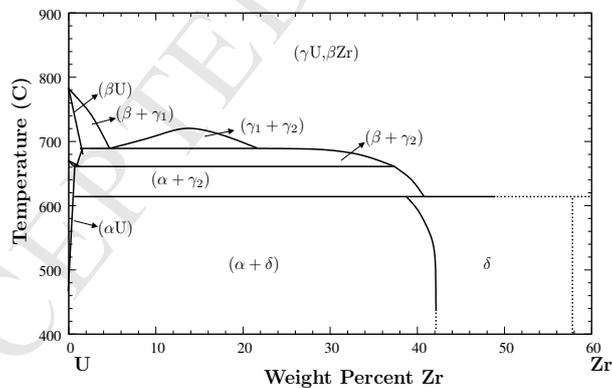


Figure 1: A sketch of the U-rich end of the U-Zr phase diagram showing phases relevant to this study [4].

45 Since the present study deals with hot extrusion of U-10Zr alloy in the $(\alpha + \delta)$ region,
 46 relevant literature on the textures developed in α -U during various thermomechanical
 47 processing conditions is briefly reviewed. It has been observed that the texture developed
 48 in α -U not only depends on the nature of the deformation (compression, tension, rolling,
 49 extrusion), but also on the temperature at which the specimen is deformed [8, 9, 10,

¹The U-10Zr alloy had $(\alpha + \delta)$ as starting microstructure [2].

50 11, 12, 13, 14]. This is due to the strong dependence of the relative importance of the
51 deformation modes on temperature (i.e., slip and twinning) in uranium [7, 12, 15]. Daniel
52 et al [7], by deforming the specimens under tension and compression, have shown that
53 at all temperatures, slip occurs most easily in the [100] direction, and depending on the
54 temperature, it would occur on the (010) plane below 500 °C, or on (001) plane at higher
55 temperatures. Slip on the (110) plane and [110] direction was observed at temperatures
56 above 150 °C. Ivanov et al [12] have observed that at rolling temperatures above 300 °C,
57 twinning becomes less pronounced while the dominant slip system is $\{110\}\langle 110\rangle$. It has
58 been pointed out that, with increasing temperature, the critical stress for activating slip
59 decreases much more rapidly than for twinning [15].

60 Previously observed deformation textures in α -U correspond, in the case of warm
61 rolling, to (010) poles aligning with the rolling direction and (001) poles aligning along the
62 normal direction to rolling [9, 14, 16], and in extruded uranium and U-2.4 wt%Nb alloy, to
63 (110) poles aligning in the extrusion direction in samples subjected to low extrusion ratio
64 and near the (310)-(100) region in samples subjected to a high extrusion ratio, respectively
65 [11, 13]. Complete loss of α texture was observed after the $\alpha \rightarrow \beta \rightarrow \gamma \rightarrow \beta \rightarrow \alpha$
66 transformation cycle while a texture memory effect was observed after the $\alpha \rightarrow \beta \rightarrow \alpha$
67 transformation cycle, the reason for which was attributed to the retention of small nuclei
68 of the parent α in the β phase ([17] and references there in). Deformation textures of the
69 intermetallic UZr₂- δ phase in U-Zr alloys, which is hexagonal, have not been published in
70 the literature and the texture development for the specific case where thermo-mechanical
71 processing is performed in the ($\alpha + \delta$) region, is not yet reported.

72 In regards to metallic uranium or any uranium based metallic alloy fuels, it was ob-
73 served that the α -U phase undergoes anisotropic irradiation growth [18]. Specifically, due
74 to the anisotropic properties of the orthorhombic crystal structure of α -U, mismatched
75 strains develop between individual grains due to their anisotropic growth during irradi-
76 ation. The stresses thus developed accumulate and can be released at grain boundaries
77 causing “tearing or cavitation swelling” [2]. Since cavitation swelling is the consequence
78 of anisotropic irradiation growth in α -U, preferred orientation of α -U grains resulting from
79 the manufacturing process plays very important role in influencing the shape changes of
80 fuel pin during irradiation. In U-Zr alloy fuel pins, cavitation swelling was observed in
81 the region where the fuel pin had ($\alpha + \delta$) microstructure [2]. However, the irradiation
82 response of monolithic δ phase is not yet known. In order to preserve the dimensional
83 stability of the fuel pins during service, the fuel material must be texture free, which
84 imposes challenges on the manufacturing process. Due to the complex interplay of phase
85 transformations, alloying element redistribution, recrystallization etc during heat treat-
86 ments, experimental in situ data is of great value to understand and ultimately predict
87 and optimize the microstructure during manufacturing [2]. In the present paper, we re-

88 port the results from our study on the texture evolution during annealing of a U-10Zr
89 alloy extruded at 600 °C, in the ($\alpha + \delta$) region in situ, using neutron diffraction. The
90 results will be used to optimize the extrusion process parameters and subsequent heat
91 treatments to obtain a microstructure, which is texture and residual stress free.

92 2 Experimental

93 Depleted uranium (DU) pieces were pickled in 25 vol% nitric acid to remove the oxide
94 layer before casting. DU pieces and crystal bar zirconium were then weighed such that
95 the alloy contained 10 wt% Zr, and melt cast in yttrium oxide crucibles under argon
96 atmosphere in a high temperature furnace with a heating rate of 20 °C/min. The alloy was
97 held isothermally for 2 hours at 1900 °C (U-10Zr composition melts at around 1400 °C)
98 after which it was cooled (i.e., furnace cooled under argon atmosphere) at a rate of about
99 20 °C/min to ambient temperature. To ensure homogeneity of the alloy, the ingot was re-
100 melted after flipping, using the same temperature cycle. The as-cast ingot had a diameter
101 of 19 mm. It was then extruded at 600 °C using a rig that was housed in a custom built
102 enclosure that has flowing argon, using a force of 306 kN. The area reduction was about
103 11.1 and the extrusion constant K was 65. Post-extrusion cooling to room temperature
104 was under flowing argon. Oxygen content after casting and extrusion was not measured
105 in the sample. Rough et al. have shown that oxygen and nitrogen impurities can make
106 α -U and α -Zr more stable than the δ phase [19]. In addition, a general feature that is
107 reported in the literature is the presence of impurity (oxygen and carbon) stabilized Zr
108 precipitates [20]. As will be shown in the subsequent section, impurity stabilized α -Zr
109 precipitates were found in the extruded alloy..

110 Texture measurement using neutron diffraction was performed on the time-of-flight
111 diffractometer HIPPO at the pulsed neutron source at LANSCE [21]. A resistive fur-
112 nace with vanadium heating elements was used to collect the diffraction patterns under
113 vacuum at high temperatures [22]. The large angular detector coverage and very high
114 neutron count rates for the instrument have been effectively used in order to optimise the
115 data collection time. Data were first acquired for 4 sample rotations (0 °, 22.5 °, 45 °, and
116 135 ° about vertical axis) and then for 3 rotations (45 °, -22.5 °, and -45 °), with 3 minutes
117 exposure time for each rotation using the 150 °, 120 °, 90 °, 60 °, and 40 ° detector banks.
118 The sample rotations along the vertical axis improve the detector coverage; specific de-
119 tails about the instrument and data collection procedure can be found in [21, 22, 23].
120 Data analysis was performed following procedures outlined in [23] using the E-WIMV
121 method to determine the orientation distribution function. Individual pole figures were
122 recalculated using MTEX software after exporting the data from MAUD (Materials Anal-
123 ysis Using Diffraction) [24]. Analysis indicated that both data sets gave similar results

124 for the texture; subsequently, 3 rotations were used for collecting the diffraction pat-
 125 terns. Diffraction patterns at non-ambient temperatures (at 200 °C during heating and
 126 at 600 °C, 650 °C, 680 °C, 700 °C, and 800 °C during heating and cooling) were collected
 127 to monitor the texture evolution as the phase transformations occur. The temperatures
 128 were selected based on the phase diagram [4] and from the differential scanning calorime-
 129 try (DSC) measurements on the as-cast alloy [25], shown in Table. 1. Texture data for
 130 the room temperature equilibrium phases, i.e., α and δ are first presented followed by the
 131 data for high temperature phases, i.e., for β (after $\alpha \rightarrow \beta$ transformation) and γ (after
 132 $\delta \rightarrow \gamma$ and $\alpha \rightarrow \beta \rightarrow \gamma$ transformations).

Table 1: Phase transformations and the associated temperatures (in °C) in U-10Zr alloy

Phase transformations [†]	$(\alpha + \delta) \rightarrow (\alpha + \gamma)$	$(\alpha + \gamma) \rightarrow (\beta + \gamma)$	$(\beta + \gamma) \rightarrow \gamma$
Phase diagram	617	662	693
DSC measurement	617.5	680 [‡]	695

[†] α : Orthorhombic; β : Tetragonal; γ : Body centered cubic; δ (intermetallic with UZr₂ stoichiometry): Hexagonal.

[‡] It is seen that the $(\alpha + \gamma) \rightarrow (\beta + \gamma)$ transformation occurred at much higher temperature than what is shown in the phase diagram. This aspect is addressed elsewhere [26].

133 3 Results

134 Fig. 2a shows the microstructure of extruded alloy, which has a lamellar microstructure
 135 consisting of α and δ -UZr₂ phases. The high density of dark contrast features correspond
 136 to the δ -UZr₂ phase while the highest contrast features, which are elongated in the ex-
 137 trusion direction (indicated by an arrow in Fig. 2a), correspond to zirconium stringers.
 138 Volume fractions of the phases were not estimated from the image. The neutron diffrac-
 139 tion pattern collected at the 150° detector bank for 22.5° sample rotation is shown in Fig.
 140 2b confirmed the presence of α and δ phases. Rietveld refinement resulted in the weight
 141 fractions of around 0.80 and 0.20 for α and δ phase, respectively, in good agreement with
 142 the U-Zr phase diagram (0.78 and 0.22 for α and δ , respectively) [4].

143 The (100), (010), (001), and (110) pole figures for α in the as-cast alloy are shown in
 144 Fig. 3a; it is seen that the texture is quite random. For the extruded alloy, the room
 145 temperature α pole figures reveal that the (100) _{α} and (110) _{α} poles are oriented along the
 146 extrusion direction (Fig. 3b). At 600 °C, within the $(\alpha + \delta)$ region, no significant change
 147 in texture is observed (Fig. 3c) while at 650 °C, in the $(\alpha + \gamma)$ region, the α texture
 148 weakens (Fig. 3d). However, after heating to 800 °C and cooling the sample back, i.e.,
 149 after the $\alpha \rightarrow \beta \rightarrow \gamma \rightarrow \beta \rightarrow \alpha$ transformation cycle, the α texture is completely lost
 150 (Fig. 3e), in accord with the observations in [13, 17].

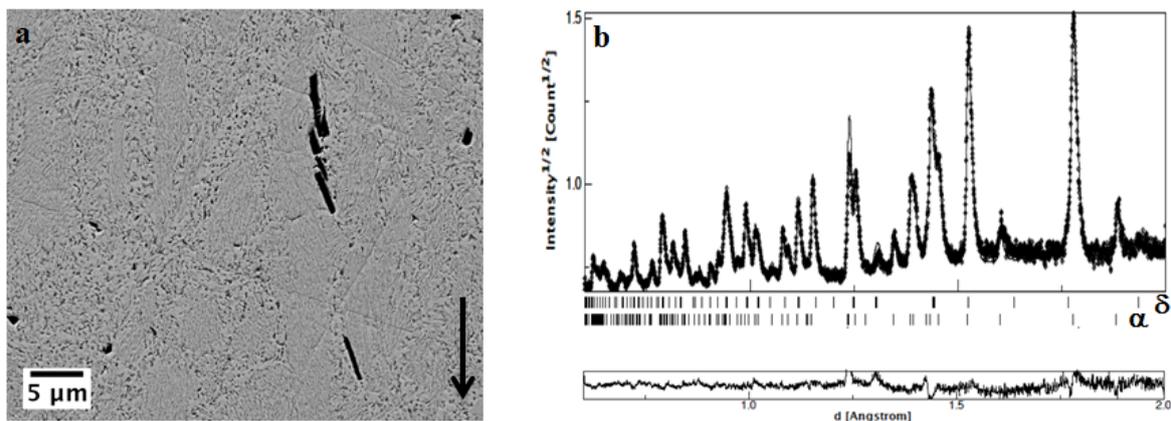


Figure 2: (a) Back scattered electron image of the extruded alloy showing two phase ($\alpha + \delta$) microstructure. The extrusion direction is indicated by an arrow. (b) Neutron diffraction pattern of the extruded alloy along with the Rietveld fit from the 150° detector bank at 22.5° sample rotation, taken at room temperature.

151 The pole figures for δ phase in the as-cast alloy are shown in Fig. 4a; it is seen
 152 that it has very weak texture, developed during casting where $(0001)_\delta$ poles are aligned
 153 parallel to the rod axis. For the extruded alloy, at room temperature, the $(0001)_\delta$ poles
 154 are oriented along the extrusion direction (Fig. 4b). This texture is preserved at 200°C
 155 and 600°C during heating. The orientation distribution with c -axes aligned along the
 156 extrusion direction starts to break up into distinct maxima with a larger spread around the
 157 extrusion direction with c -axis maxima even observed approximately radially at 600°C
 158 (Fig. 4c and Fig. 4d, respectively).

159 After heating the sample to 800°C and cooling back, i.e., after the $\delta \rightarrow \gamma \rightarrow \delta$
 160 transformation cycle, the texture was analysed at 600°C and the pole figures are shown
 161 in Fig. 4e. It is seen that the $(0001)_\delta$ texture is preserved indicating a memory effect.
 162 Comparing the pole figures at room temperature and after the thermal cycle (Fig. 4b
 163 and 4e), spottiness is observed, suggesting the presence of few large grains.

164 The pole figures for β phase at 680°C , after the $\alpha \rightarrow \beta$ transformation, show that
 165 it has a random texture (Fig. 5a). Even though no conclusive orientation relationship
 166 for the $\alpha \rightarrow \beta$ transformation is known [17], several possible variants are likely and the
 167 relatively weak α texture (max. of ~ 2.5 multiples of random distribution) results in a
 168 random texture of the β phase.

169 The γ phase texture is shown at 650°C in the $(\alpha + \gamma)$ phase field after the partial
 170 $\delta \rightarrow \gamma$ transformation, 700°C , after the partial $\beta \rightarrow \gamma$ transformation at which point the
 171 alloy is completely in the γ phase, 800°C during heating, and at 700°C during cooling
 172 in Fig. 5b, 5c, 5d, and 5e, respectively. It is pointed out that though the diffraction
 173 peaks for γ phase completely overlap with those of α , the texture of γ , though weak, is

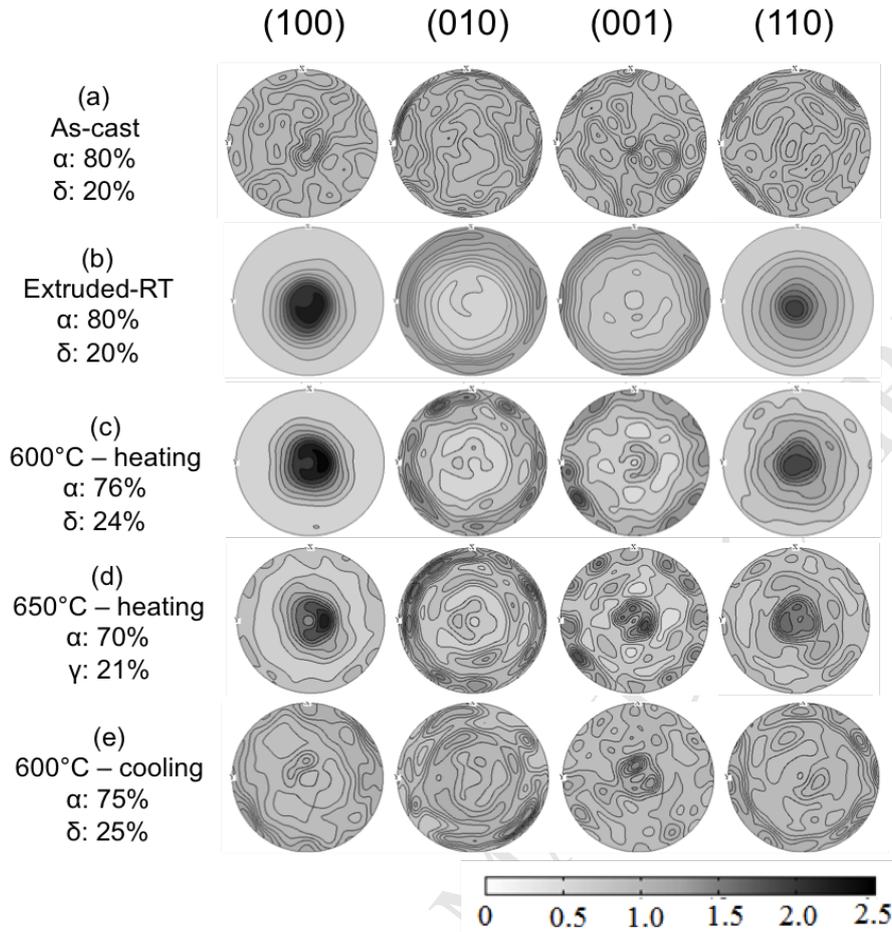


Figure 3: Pole figures of α -U (a) of as-cast alloy (b) Extruded alloy at room temperature (RT) (c) at 600 °C during heating (d) at 650 °C during heating (e) at 600 °C during cooling. The phases along with their weight fractions present at each temperature from the Rietveld refinement are also shown. At 650 °C, 9% of β -U was found to be present. Extrusion direction is at the centre.

174 captured quite nicely at 650 °C (in the $(\alpha + \gamma)$ region). It is seen that $(110)_\gamma$ and $(111)_\gamma$
 175 poles are aligned in the extrusion direction. This becomes quite clear when compared
 176 with the texture, which is stronger, obtained at 700 °C and 800 °C, where the sample has
 177 100% γ phase (Fig. 5c, 5d, and 5e).

178 4 Discussion

179 In the present study, the preferred orientation developed in α -U after extrusion is in
 180 agreement with the ones reported in literature [11, 13]. While the complete loss of α
 181 texture after the $\alpha \rightarrow \beta \rightarrow \gamma \rightarrow \beta \rightarrow \alpha$ transformation cycle observed in this study
 182 in situ by capturing the texture changes in various phase fields is in accord with the
 183 published literature (note, in previous studies these were performed ex situ at room

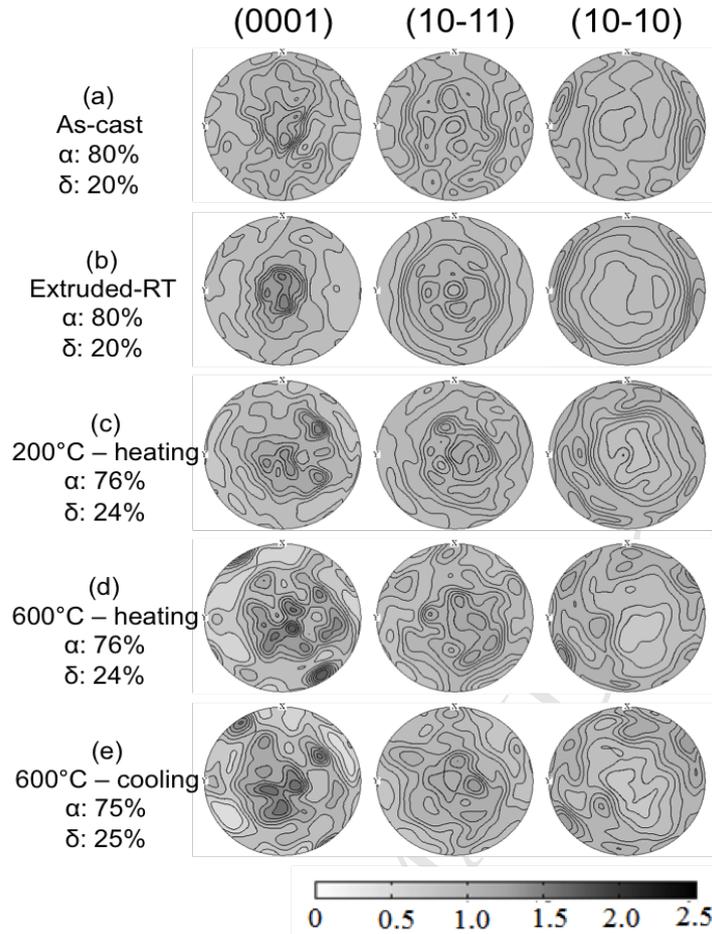


Figure 4: Pole figures of δ -UZr₂ (a) in the as-cast alloy (b) Extruded alloy at room temperature (RT) (c) at 200 °C (d) at 600 °C during heating (e) at 600 °C during cooling. The pole figures are plotted to the same scale as in α . The phases along with their weight fractions present at each temperature obtained from the Rietveld refinement are also shown. Extrusion direction is at the centre.

184 temperature, after the transformation cycle), perhaps the most interesting result is the
 185 texture memory effect observed for the δ phase.

186 Basak et al [27] have shown that δ phase forms from the high temperature γ phase via
 187 ω transformation mechanism and is related to γ by the following orientation relationship
 188 (OR): $(111)_{\gamma} \parallel (0001)_{\delta}$ and $\langle \bar{1}10 \rangle_{\gamma} \parallel \langle 11\bar{2}0 \rangle_{\delta}$. Specifically, the δ phase forms by the
 189 collapse of alternate $(111)_{\gamma}$ planes in opposite direction with the third plane unaltered to
 190 form the intermediate $(0001)_{\delta}$ plane, followed by the partial ordering of U and Zr atoms
 191 in the hexagonal cell and has four possible variants.

192 DSC data for U-10Zr alloy published elsewhere have shown the following [25]: (a) from
 193 the cooling curve, the high temperature γ phase first transforms partially to α at 663 °C
 194 and the remnant γ then transforms to δ at 616 °C producing the $(\alpha + \delta)$ microstructure
 195 at room temperature and (b) from the heating curve, the $\delta \rightarrow \gamma$ transformation occurs

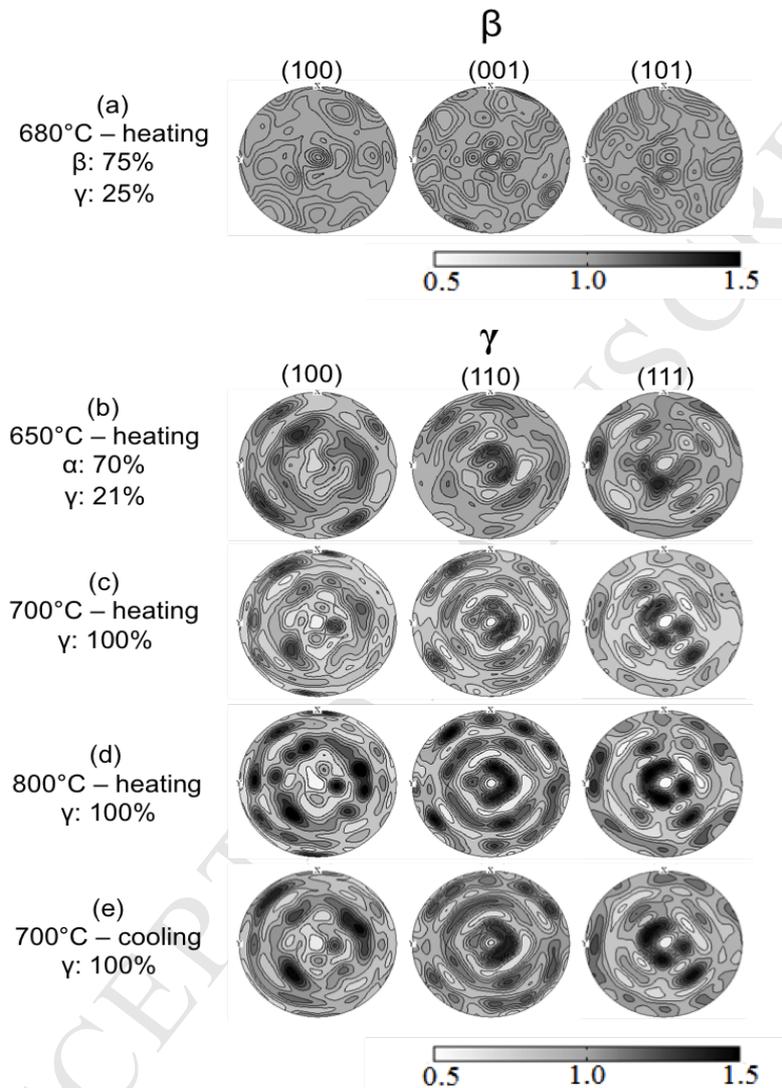


Figure 5: Pole figures of extruded alloy for (a) β -U at 680 °C (b) γ -U at 650 °C during heating (c) γ -U at 700 °C during heating (d) γ -U at 800 °C during heating (e) γ -U at 700 °C during cooling. The phases along with their weight fractions present at each temperature from the Rietveld refinement are also shown. At 650 °C, 9% of β -U was found to be present. Extrusion direction is at the centre.

196 at 617.5 °C. As described above, during cooling, the $\gamma \rightarrow \delta$ transformation at 616 °C
 197 happens by the collapse of $(111)_\gamma$ planes to form $(0001)_\delta$ planes.

198 The microstructural development observed in the present study can be rationalized in
 199 the following way. For U-10Zr alloy, while heating, the following transformations occur
 200 as per the equilibrium phase diagram: from the room temperature ($\alpha + \delta$) with ~34
 201 vol% δ and 66 vol% α , the δ phase first transforms to γ at 617 °C. The α transforms
 202 to β at 662 °C and at 693 °C, β transforms to γ . The alloy, when it is 100% γ (i.e., at
 203 700 °C), will contain some fraction (i.e., 34 vol%) resulting from $\delta \rightarrow \gamma$ and the rest (i.e.,
 204 66 vol%) from $\alpha \rightarrow \beta \rightarrow \gamma$. So, when the alloy is extruded in the ($\alpha + \delta$) region, one
 205 would expect the texture developed for the δ phase and the one for the γ phase after
 206 $\delta \rightarrow \gamma$ transformation to be related by the OR: $(0001)_\delta \parallel (111)_\gamma$. Comparing the pole
 207 figures of $(0001)_\delta$ at 600 °C and $(111)_\gamma$ at 650 °C (Fig. 4d and 5b respectively), the OR
 208 is not clearly reflected.

209 When the alloy is 100% γ , the texture is much stronger than it is at 650 °C (Fig.
 210 5b, c, d, and e) and can be interpreted in the following way: the high temperature γ
 211 (i.e., at temperatures >700 °C where it is 100% γ) forms, as mentioned above, from two
 212 different transformation paths; some as a result of $\delta \rightarrow \gamma$ and the rest via $\alpha \rightarrow \beta \rightarrow \gamma$
 213 transformation. So, the texture of γ should reflect the OR with δ and an OR with β (i.e.,
 214 after $\alpha \rightarrow \beta \rightarrow \gamma$). However, β texture is quite random (Fig. 5a) and an OR between
 215 β and γ has not been established yet, mainly because of the difficulty in retaining both
 216 phases simultaneously. From Fig. 5c, d, and e, it is seen that $(110)_\gamma$ and $(111)_\gamma$ poles are
 217 aligned along the extrusion direction. While the $(111)_\gamma$ texture suggests the presence of
 218 an OR with δ (i.e., $(111)_\gamma \parallel (0001)_\delta$), the matching is not sufficient to claim its presence.
 219 The $(110)_\gamma$ texture does not seem to be related to either β or α .

220 The texture memory of δ phase after the temperature cycle suggests that only those
 221 γ grains that have formed as a result of $\delta \rightarrow \gamma$ during heating have again transformed
 222 to δ during cooling. Because of the relatively moderate starting texture of δ phase, and
 223 the added complexity of phase transformations involving α and β at high temperatures
 224 (i.e., $\alpha \rightarrow \beta \rightarrow \gamma$), the OR between δ and γ was not clearly reflected in the pole figures.
 225 A similar experiment on a deformed U-50Zr alloy, which contains 100% δ from room
 226 temperature to around 600 °C, would help clarify the texture memory effect as this alloy
 227 composition only undergoes $\delta \rightarrow \gamma$ transformation. Since the preferred orientation of α
 228 phase is lost after a phase transformation cycle (i.e., upon $\alpha \rightarrow \beta \rightarrow \gamma \rightarrow \beta \rightarrow \alpha$), studies
 229 on the possibility of randomizing the δ texture by suitable heat treatments and on the
 230 irradiation response of δ phase for possible anisotropic swelling effects need to be carried
 231 out.

232 Conclusions

233 In summary, we have carried out an in situ neutron diffraction study to monitor the
234 texture evolution during annealing in a U-10Zr alloy hot extruded in ($\alpha + \delta$) region. The
235 results show a complete loss of α texture and suggest a texture memory effect for the δ
236 phase after the $\alpha \rightarrow \beta \rightarrow \gamma \rightarrow \beta \rightarrow \alpha$ and $\delta \rightarrow \gamma \rightarrow \delta$ transformation cycles, respectively.

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