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Transformation behavior of the γ U(Zr,Nb) phase under continuous cooling conditions

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ABSTRACT

The selected alloy for the fabrication process of a high-density monolithic-type nuclear fuel with U-Zr-Nb alloys as meat and Zry-4 as cladding, has to remain in the γ U(Zr,Nb) phase during the fabrication process. The aim is to define a range of concentrations in which the γ U(Zr,Nb) phase doesn't decompose under this process conditions.

In this work, several U alloys with concentrations between 13.9-43.7 wt.%Zr and 0-7.3 wt.% Nb were fabricated. The γ U(Zr,Nb) \rightarrow δ UZr₂ and γ U(Zr,Nb) \rightarrow α U transformations are being evaluated by thermo-electrical resistivity measurements under different continuous cooling conditions (between 4-120 °C/min).

For a cooling rate of 4°C/min a linear regression was determined by fitting the values for the start decomposition temperature as a function of Nb concentration. Under these conditions, a concentration of 23 wt% Nb would be enough to avoid any transformation of the γ U(Zr,Nb) phase. In experiences involving higher cooling conditions, it has been determined that this concentration can be reduced around fifty percent.

INTRODUCTION

Since 1978, the international RERTR program has promoted the reduction of the enrichment for the U alloys used as fuels in MTR-type reactors. The concentration of the isotope ²³⁵U shouldn't exceed the 20 at%. This requirement demands an increase in the U density in the fuel. Considering the existing fabrication process for dispersed type fuels with aluminide, oxide and silicide it's possible to convert most of the functioning MTR-type reactors. However, these compounds are not dense enough to fulfill the high flux reactor needed densities [1, 2, 3, 4]. For this purpose, several alloys with higher U densities are under study. It's mandatory that these alloys retain the bcc γ U phase, because its good behavior under irradiation [4, 5].

Internationally, U-Mo alloys are under study for the qualification of dispersed and monolithic-type fuel element. In Argentina, the U-Zr and U-Zr-Nb alloys are also under study as an alternative for the fabrication of a monolithic-type fuel U(Zr,Nb)/Zry-4. As first step, a set of alloys with U densities between 7-10 gU/cm³ have been studied.

From the binary equilibrium phase diagram U-Zr it can be seen that the $\gamma\text{U}(\text{Zr})$ phase is stable above 800°C for U-Zr alloys with Zr concentration below 50 wt%. It also shows that δUZr_2 phase (hP3) and the αU phase (oC4) are the stable phases at room temperature [6]. The δUZr_2 is formed through the allotropic reaction $\gamma\text{U}(\text{Zr}) \rightarrow \delta\text{UZr}_2$ for temperatures near 610°C, while the αU phase is formed by precipitation. Dynamic experiences results related with this binary system are only presented by Bauer in 1959 [7]. In this work is discussed, based on the thermo-electrical relative resistivity vs Temperature (ρ_{rel} vs T) curves, a possible δUZr_2 phase rearrangement of the crystal structure.

According to the U-Zr-Nb ternary system, the experimental results presented by Dwight and Mueller in [9], show that the phase $\gamma\text{U}(\text{Zr,Nb})$ is stable for the concentration range surrounding the existence of the δUZr_2 phase at temperatures higher than 700°C. Also, the solubility of Nb in the δUZr_2 phase is lower than 10 wt%. The $\gamma\text{U}(\text{Zr,Nb})$ phase could be retained as metaestable depending on the cooling rate, the Nb and Zr concentrations [4, 8, 9].

The aim of this work is to define a concentration domain in which the $\gamma\text{U}(\text{Zr,Nb})$ phase can be retained as metaestable considering the thermal evolution experienced for the U-base alloy during the fabrication process of a monolithic-type miniplate U(Zr,Nb)/Zry-4.

To study the behavior of the $\gamma\text{U}(\text{Zr,Nb})$ phase, ten alloys with concentrations ranging from 13.9 to 43.7 wt.% Zr and from 0 to 7.3 wt.% Nb, were fabricated (U densities 7-10 g/cm³). As a first stage, all the alloys were isothermally treated at 850 °C during 1 h or 24 h and quenched (quenching in cold water will be considered as the fastest cooling rate). X-Ray Diffraction, Optical Microscopy and Scanning Electron Microscopy techniques were employed to perform the morphological and crystallographic characterization. The $\gamma\text{U}(\text{Zr,Nb}) \rightarrow \delta\text{UZr}_2$ allotropic reaction and the αU precipitation were evaluated by measurements of thermo-electrical resistivity under different continuous cooling conditions (between 4-120 °C/min).

EXPERIMENTAL PROCEDURE

Ten different U alloys were fabricated with concentrations ranging from 13.9 to 43.7 wt% of Zr and from 0 to 7.3 wt% of Nb. The alloying elements employed were: depleted U (0.2 at% of ²³⁵U, principal impurities: 27 ppm Fe, 60 ppm Mg y 24 ppm Si), Zr (principal impurities: 420 ppm O₂ y <170 ppm Fe) and Nb (principal impurities: 50 ppm O₂ y <200 ppm Fe). Table 1 shows the concentration for the ten alloys fabricated.

Sample	U (wt%)	Zr (wt%)	Nb (wt%)
1	56.3	43.7	0.0
2	56.2	42.0	1.8
3	61.6	32.0	6.4
4	66.8	33.2	0.0
5	66.6	30.7	2.7
6	66.1	27.1	6.8
7	75.3	24.7	0.0
8	74.9	21.6	3.5
9	74.5	18.2	7.3
10	81.9	13.9	4.2

Table 1: Concentration of the fabricated alloys.

These alloys were fabricated by arc-melting under argon atmosphere in a water cooled copper crucible using a non-consumable tungsten electrode.

Isothermal treatments at 850°C

The samples were sealed in quartz tubes under argon atmosphere; isothermal heat treatments at 850 °C were performed in two independent stages (1 h or 24 h) and quenched without tube breaking.

Phase characterization was performed by X-Ray Diffraction (XRD – Philips PW3710), Optical Microscopy (OM – Olympus BX60M) and Scanning Electron Microscopy (SEM - FEI - QUANTA 200). Samples were suitably grinded and mechanically polished up to 1 μm diamond paste. In particular for OM, a final chemical etching with a solution $\text{HNO}_3:\text{HF}:\text{H}_2\text{O}$ in volumetric proportion 47:3:50 was made. XRD measurements were performed at room temperature with filtered Cu $K\alpha$ radiation, being 26 ° and 136 ° the start and end position respectively and 1 s the scan step time. Powder diffraction technique was applied to bulk samples. Crystalline structure identification and the estimation of the lattice parameters were obtained using the PowderCell program [10].

Continuous cooling experiments

Phase transformations in continuous cooling conditions were evaluated by measurements of thermo-electrical resistivity.

A first set of experiences involved 3 steps: continuous heating at a rate of 4°C/min up to 900°C, an isothermal stage for 1 minute at 900°C and continuous cooling at a rate of 4°C/min up to 200°C. A second set of experiences involved also 3 steps: continuous heating at a rate of 120°C/min up to 900°C, an isothermal stage for 1 minute at 900°C and continuous cooling at a rate of X°C/min (X=4, 8, 15, 30, 60 and 120). This second set of experiences was performed under an argon flux to extend the linear range of controlled cooling.

Samples dimensions for both sets of experiences were 20x1x0.5 mm.

RESULTS & DISCUSSION

Isothermal treatments at 850°C

The phases identified by XRD on each alloy after both isothermal treatments at 850°C are presented in Table 2.

Samples	1h	24h
1	δUZr_2	δUZr_2
2	$\gamma\text{U}(\text{Zr,Nb}) + \delta\text{UZr}_2$	$\gamma\text{U}(\text{Zr,Nb}) + \delta\text{UZr}_2$
3	$\gamma\text{U}(\text{Zr,Nb})$	$\gamma\text{U}(\text{Zr,Nb})$
4	δUZr_2	$\gamma\text{U}(\text{Zr,Nb}) + \delta\text{UZr}_2$
5	δUZr_2	$\gamma\text{U}(\text{Zr,Nb}) + \delta\text{UZr}_2$
6	$\gamma\text{U}(\text{Zr,Nb})$	$\gamma\text{U}(\text{Zr,Nb})$
7	$\delta\text{UZr}_2 + \alpha\text{U}$	$\delta\text{UZr}_2 + \alpha\text{U}$
8	$\gamma\text{U}(\text{Zr,Nb}) + \alpha\text{U}$	$\gamma\text{U}(\text{Zr,Nb}) + \alpha\text{U}$
9	$\gamma\text{U}(\text{Zr,Nb}) + \alpha\text{U}$	$\gamma\text{U}(\text{Zr,Nb}) + \alpha\text{U}$
10	$\gamma\text{U}(\text{Zr,Nb}) + \alpha\text{U}$	$\gamma\text{U}(\text{Zr,Nb}) + \alpha\text{U}$

Table 2: Phase identification after heat treatments at 850°C.

The only phases identified are: $\gamma\text{U}(\text{Zr,Nb})$, δUZr_2 and αU . Samples 3 and 6 were the only two alloys that retained the $\gamma\text{U}(\text{Zr,Nb})$ phase as metaestable after quenching. The alloys with lower Zr concentrations (samples 7, 8, 9 and 10), showed a partial precipitation of the αU phase. In sample 7, the remaining $\gamma\text{U}(\text{Zr,Nb})$ transforms into δUZr_2 by the peritectoid reaction: $\gamma\text{U}(\text{Zr,Nb}) + \alpha\text{U} \rightarrow \delta\text{UZr}_2$. In the alloys with higher Zr concentration (samples 1, 2, 4 and 5), the $\gamma\text{U}(\text{Zr,Nb})$ transforms partially/completely to δUZr_2 as an allotropic reaction.

Figure 1 shows the XRD phase identification performed on samples 1, 2, 6 and 8 as an example of the four different situations explained in the previous paragraph. Lattice parameters estimated for the

$\gamma\text{U}(\text{Zr,Nb})$ phase are shown in Table 3. The ones proposed by Dwight and Mueller in [9] are include for comparison purposes.

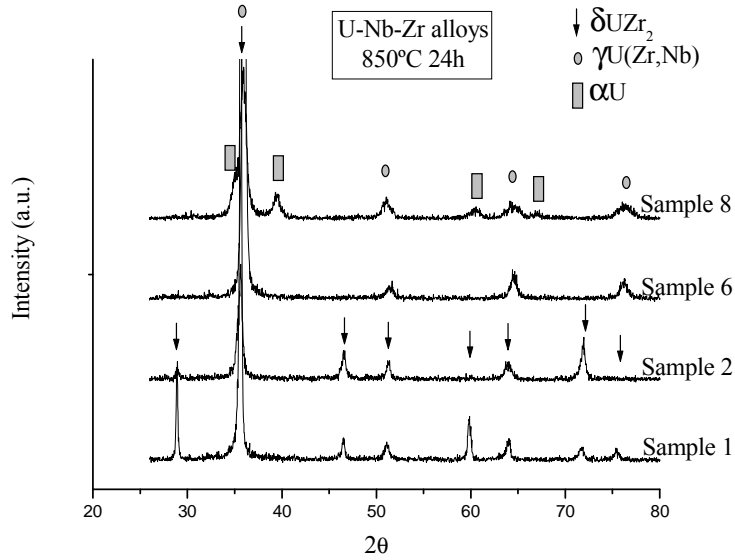


Figure 1: Phase identification by XRD for samples 1, 2, 6 and 8. Heat treatment 850°C – 24h.

Sample	Isothermal treatment 850 °C		Dwight and Mueller in [8]
	a_{1h} (Å)	a_{24h} (Å)	a_D (Å)
2	3.578	3.558	3.563
3	3.535	3.535	3.525
4	-	3.568	3.545
5	-	3.535	3.535
6	3.537	3.527	3.515
8	3.535	3.538	3.515
9	3.527	3.517	3.50
10	3.508	3.503	3.50

Table 3: Lattice parameter for the $\gamma\text{U}(\text{Zr,Nb})$ phase.

Due to peak overlapping of the $\gamma\text{U}(\text{Zr,Nb})$ phase with the ones corresponding to the δUZr_2 phase, the partial/total allotropic reaction $\gamma\text{U}(\text{Zr,Nb}) \rightarrow \delta\text{UZr}_2$ was corroborated by OM. An example of the partial transformation is shown in Figure 2. The $\gamma\text{U}(\text{Zr,Nb})$ can be observed as a dark grey phase and the δUZr_2 phase is identified as the light grey one.

Figure 3 shows a SEM image obtained for sample 1 on which a needle-shape precipitate is observed. However, δUZr_2 was the only phase identified by XRD. Comparing this precipitation morphology with micrographs presented in [11], this metallographic component could be associated with the αZr phase. The absence of this phase in Table 2 is explained by its low volumetric amount, lower than the detection limit for XRD technique.

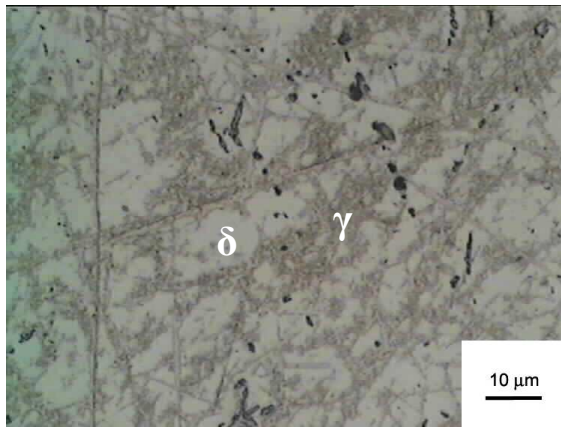


Figure 2: Sample 2-850°C-24h, partial allotropic reaction $\gamma\text{U}(\text{Zr,Nb}) \rightarrow \delta\text{UZr}_2$. OM image.

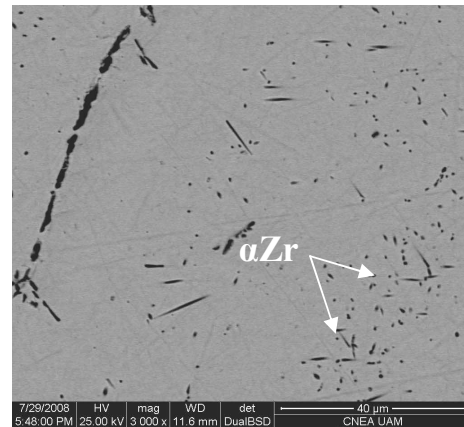


Figure 3: Sample 1-850°C-24h-SEM image.

Continuous cooling experiments 4°C/min

The two reactions described in the previous section were identified in these experiences. In Figure 4, the complete allotropic reaction $\gamma\text{U} \rightarrow \delta\text{UZr}_2$ (sample 1, Figure 4a) and the αU precipitation (sample 8, Figure 4a) are presented as example. In Figure 4b is presented the ρ_{rel} vs T curve when both reactions occur (sample 7, Figure 4b).

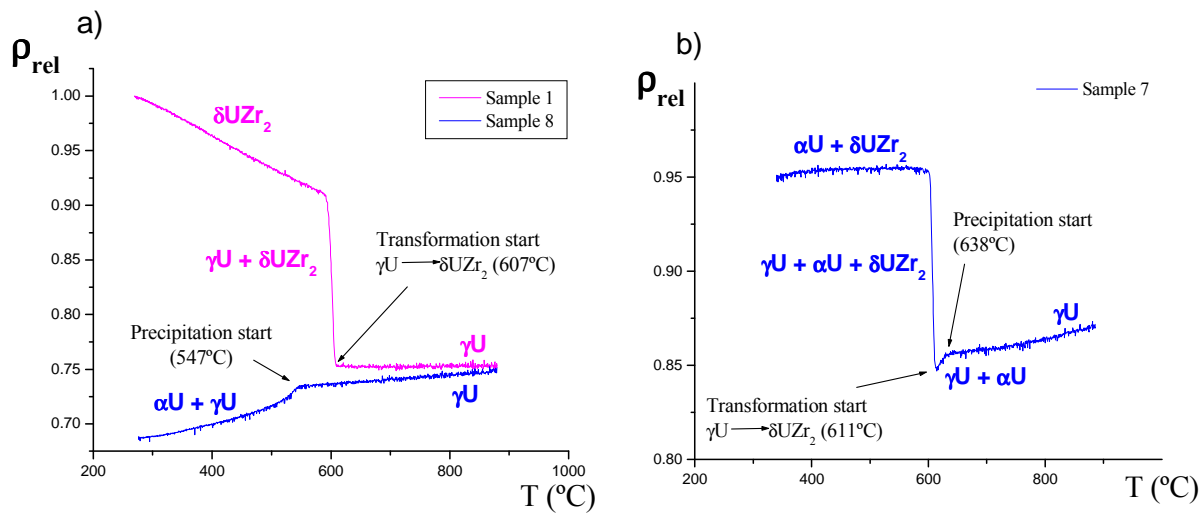


Figure 4: ρ_{rel} vs T curves for a cooling rate of 4 °C/min a) Allotropic reaction $\gamma\text{U} \rightarrow \delta\text{UZr}_2$ in sample 1 and αU precipitation in sample 8. b) Both reactions in sample 7.

For sample 1 (U-43.7wt% Zr) it's observed that at temperatures higher than 607°C the values for the ρ_{rel} is practically constant with the temperature. Between 607 and 591°C, the ρ_{rel} increases abruptly with temperature. Below 591°C, ρ_{rel} grows linearly in cooling with a slope much lower than the one observed between (607 – 591) °C. Being sample 1 a U-Zr binary alloy and knowing the equilibrium binary phase diagram U-Zr [6] it's possible to associate every temperature range with monophasic or biphasic domains. Hence, for sample 1, at temperatures higher than 607 °C, γU is the only phase present in this alloy. The $\gamma\text{U} \rightarrow \delta\text{UZr}_2$ allotropic reaction starts at 607°C and completes at 591°C. Finally, at temperatures lower

than 591°C, δUZr_2 is the only phase present in the alloy. This result reproduces what was observed for the transformation $\gamma\text{U} \rightarrow \delta\text{UZr}_2$ in [7].

The start and finish temperatures for the allotropic reaction $\gamma\text{U} \rightarrow \delta\text{UZr}_2$ are a little lower than the ones observed in the binary diagram U-Zr [6]. A similar analysis can be made for sample 8 (Figure 4a). In this case, the beginning of the αU precipitation reaction occurs at 547 °C.

Hence, from now on, an abrupt increment of the ρ_{rel} value in cooling will be associated with the allotropic $\gamma\text{U} \rightarrow \delta\text{UZr}_2$ reaction and a decrease in the slope of the curve ρ_{rel} vs T will be associated with the precipitation of the αU phase.

All the samples on which the allotropic $\gamma\text{U}(\text{Zr,Nb}) \rightarrow \delta\text{UZr}_2$ reaction was identified (samples 1-5 and 7) are shown in Figure 5a. For the binary U-Zr alloys (samples 1, 4 and 7) the beginning of the $\gamma\text{U}(\text{Zr,Nb})$ decomposition is estimated between 607 and 611 °C. These temperatures are in concordance with temperature informed in the binary equilibrium diagram. The increment of the Nb concentration in the U-Zr alloy decreases the start temperature reaction. Hence, the thermal range in which the $\gamma\text{U}(\text{Zr,Nb})$ phase is retained increases. As observed in Figure 5a, a concentration of 1.8 wt% Nb (sample 2) decreases the start temperature reaction ~ 50 °C comparing with the binary alloys. A concentration of 2.7 wt% Nb (sample 5) decreases this temperature ~ 75 °C and 6.4 wt% Nb (sample 3) decreases this temperature more than 200 °C. The alloys 6 and 9 showed experimental deficiencies and therefore, their ρ_{rel} vs T curves aren't considered.

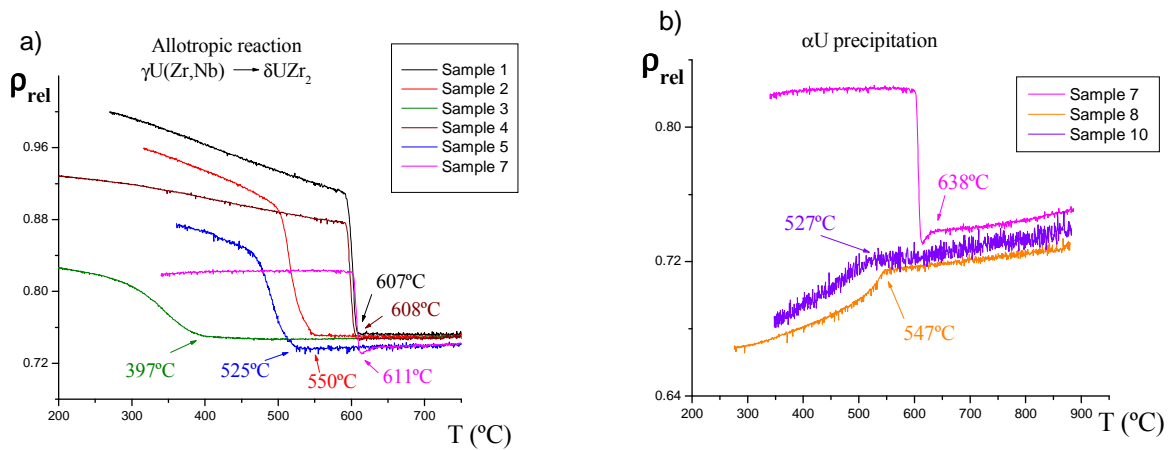


Figure 5: ρ_{rel} vs T curves for a cooling rate of 4 °C/min. Start temperature reaction vs wt% Nb a) Allotropic $\gamma\text{U}(\text{Zr,Nb}) \rightarrow \delta\text{UZr}_2$ Reaction. b) αU precipitation.

The same tendency is observed for the αU precipitation, Figure 5b. The αU precipitation start temperature was estimated in 638 °C for the binary U-Zr (sample 7). A concentration of 3.5 wt% Nb (sample 8) decreases this temperature in ~ 90 °C. A concentration of 4.2 wt% Nb, (sample 10) decreases this temperature in ~ 110 °C.

In Table 4 are summarized the transformation temperatures. $T_s^{\gamma \rightarrow \delta}$ indicates the start and $T_F^{\gamma \rightarrow \delta}$ indicates the final of the allotropic $\gamma\text{U}(\text{Zr,Nb}) \rightarrow \delta\text{UZr}_2$ reaction. $T_s^{\gamma \rightarrow \alpha}$ indicates the start of the αU precipitation.

The temperatures estimated included in Table 4 are plotted in two pseudo ternary U+Zr-Nb diagrams grouping them by reaction type, Figure 6. The alloys with U concentration ([U]) ranging from 75.3-81.9 wt% show αU precipitation (Figure 6a) and the alloys with [U] between 56.3-75.3 wt% show the allotropic reaction $\gamma\text{U}(\text{Zr,Nb}) \rightarrow \delta\text{UZr}_2$ (Figure 6b). Both graphics include a linear regression that represents the *solvus* curve for each reaction. Each linear regression is described by the following equations:

$$T_I^{\gamma \rightarrow \delta} = 609.5^\circ\text{C} - 32.9^\circ\text{C}/\% [\text{Nb}]$$

$$T_F^{\gamma \rightarrow \delta} = 591.4^\circ\text{C} - 51.1^\circ\text{C}/\% [\text{Nb}]$$

$$T_I^{\gamma \rightarrow \alpha} = 638.1^\circ\text{C} - 26.3^\circ\text{C}/\% [\text{Nb}]$$

Sample	Reaction	T_S (°C)	T_F (°C)
1	$\gamma\text{U}(\text{Zr},\text{Nb}) \rightarrow \delta\text{UZr}_2$	607	591
2		550	492
3		397	265
4		608	592
5		525	456
7	$\gamma\text{U}(\text{Zr},\text{Nb}) \rightarrow \delta\text{UZr}_2$	611	595
	αU precipitation	638	-
8	αU precipitation	547	-
10		527	-

Table 4: Temperatures estimated for the reaction's start.

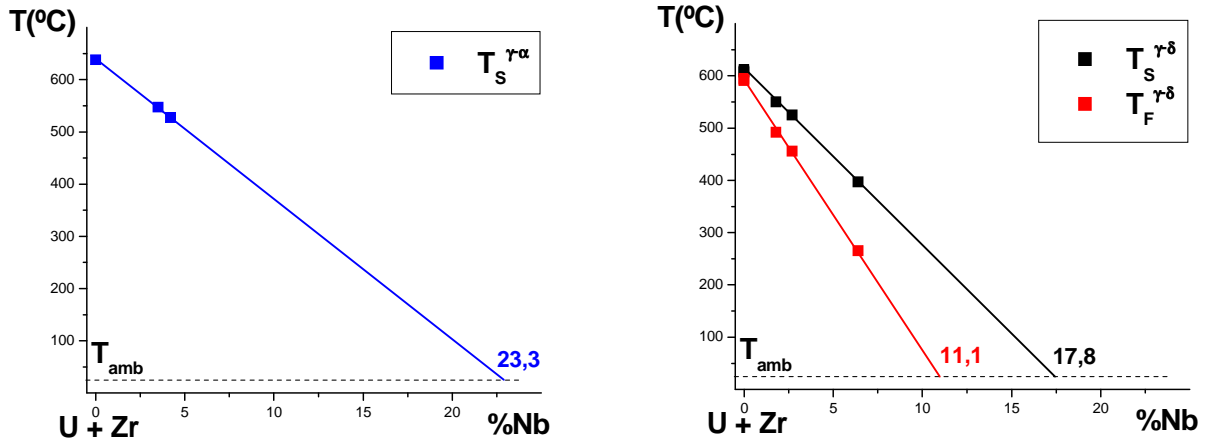


Figure 6: Start/finish temperatures reaction in pseudo binary (U+Zr)-Nb. a) αU precipitation, $75.3 < [\text{U}] < 81.9$
b) Allotropic $\gamma\text{U}(\text{Zr},\text{Nb}) \rightarrow \delta\text{UZr}_2$ reaction, $56.3 < [\text{U}] < 75.3$

Extrapolating these equations up to room temperature it's possible to predict that ~23 wt% Nb is enough to avoid αU precipitation and ~18 wt% Nb is enough to avoid the allotropic reaction $\gamma\text{U}(\text{Zr},\text{Nb}) \rightarrow \delta\text{UZr}_2$. As a consequence, 23 wt% Nb in a ternary alloy U-Zr-Nb would be enough to retain the desired $\gamma\text{U}(\text{Zr},\text{Nb})$ phase under a continuous cooling rate of 4°C/min.

Continuous cooling experiments 4-120°C/min

The technological interest is set in the concentration domain surrounding alloy 3, since this alloy retained the $\gamma\text{U}(\text{Zr},\text{Nb})$ phase under quenching. In this region, the possibility of retain as metaestable the $\gamma\text{U}(\text{Zr},\text{Nb})$ phase depends on avoiding the allotropic $\gamma\text{U}(\text{Zr},\text{Nb}) \rightarrow \delta\text{UZr}_2$ reaction under different continuous cooling conditions. This is why the analysis will be focused in the behavior of this allotropic reaction under different cooling rates conditions. Since samples 8-10 only show αU precipitation, aren't considered for this analysis.

The experimental conditions are insufficient to determine precisely the transformation temperature. As a consequence, start and finish temperatures values for this reaction aren't considered. The maximum

cooling rate considered (120°C/min) has been selected accordingly cooling rates measured during the steps of lamination involved in the fabrication process of a monolithic miniplate.

As mentioned before, the binary U-Zr alloys (samples 1, 4 and 7) show the product of the allotropic reaction when quenched from 850 °C. It's expected then; that for all the cooling rates studied, this reaction occurs. However, no significant change in the start temperature values for this reaction is observed. As an example, the curves ρ_{rel} vs T under all the continuous cooling conditions studied corresponding to the sample 4 are presented, (Figure 7a).

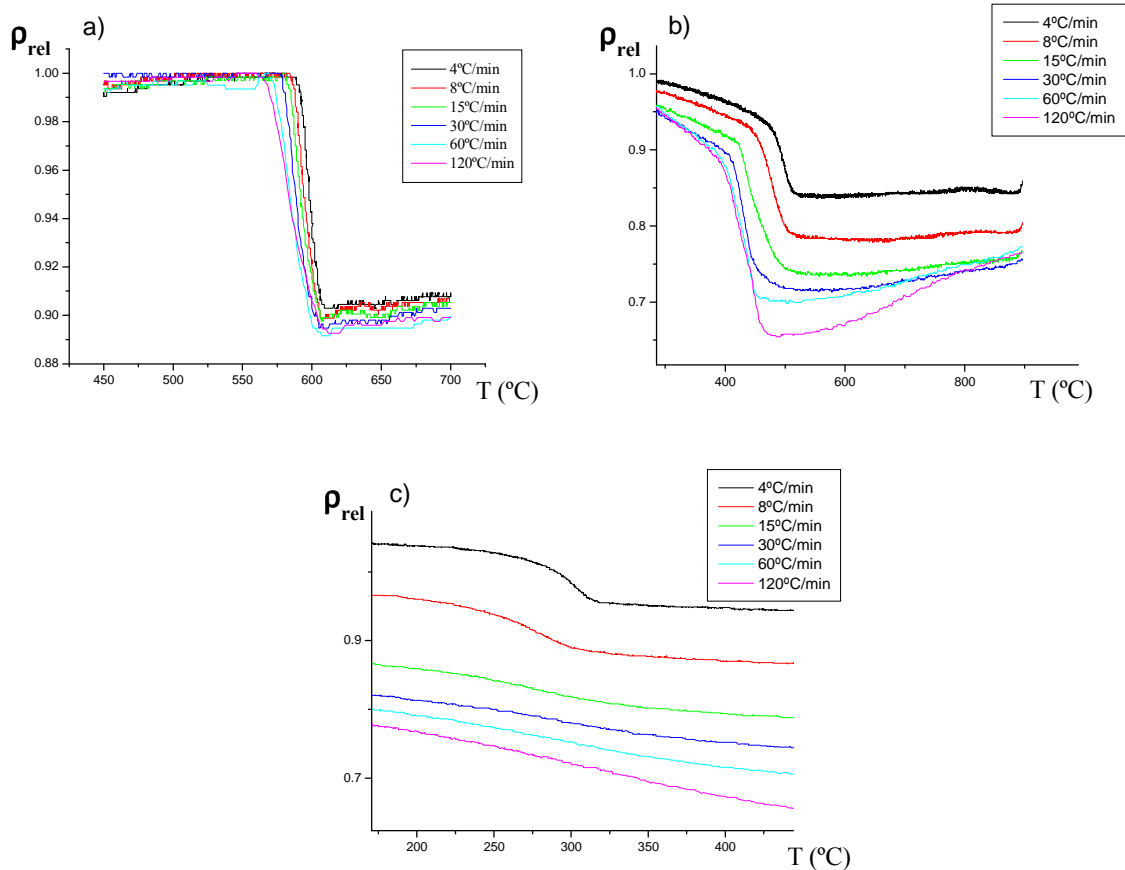


Figure 7: ρ_{rel} vs T curves for different cooling rates. a) sample 4, b) sample 5 and c) sample 3

In those alloys with Nb concentration around 2-3 wt% (samples 2 and 5) the allotropic reaction is also observed for all the cooling rates performed. However, a remarkable decrease in the start temperature's values for this reaction is observed, (sample 5-Figure 7b).

Finally, with Nb concentration around 6-7 % (samples 3 and 6) the allotropic reaction is clearly observable at the lowest cooling rates (4-8 °C/min). In cooling rates between 30-120°C/min this reaction doesn't occur, hence, the $\gamma U(Zr,Nb)$ phase is retained. The results at 15°C/min doesn't allow a conclusion in this sense. As an example, the results for sample 3 are shown, Figure 7c.

As a consequence, in continuous low rates controlled cooling (4°C/min, Figure 6), it was observed that ~17 wt% Nb would be enough to retain the $\gamma U(Zr,Nb)$ phase up to room temperature. The lowest cooling rate considering the thermal evolution experienced for the U-base alloy during the fabrication process of a monolithic-type miniplate U(Zr,Nb)/Zry-4 is around 120°C/min. In Figure 7c can be observed that 6.4 wt% Nb would be enough to retain the $\gamma U(Zr,Nb)$. As a consequence in experiences involving higher

cooling conditions, the concentration needed to avoid the $\gamma\text{U}(\text{Zr,Nb})$ decomposition would be reduced a fifty percent comparing with the ones obtained for 4°C/min.

CONCLUSIONS

To study the behavior of the $\gamma\text{U}(\text{Zr,Nb})$ phase, ten alloys with concentrations ranging from 13.9 to 43.7 wt.% Zr and from 0 to 7.3 wt.% Nb, were fabricated (U densities 7-10 g/cm³).

The results from the isothermal treatments at 850 °C allowed the identification of only three phases: $\gamma\text{U}(\text{Zr,Nb})$, δUZr_2 and αU . Sample 3 (U-32.0 wt% Zr-6.4 wt% Nb) and sample 6 (U-27.1 wt% Zr-6.8 wt% Nb) were the only two alloys that retained the $\gamma\text{U}(\text{Zr,Nb})$ phase as metaestable by quenching (fastest cooling rate).

The measurements of thermo-electrical resistivity allowed the identification of the $\gamma\text{U}(\text{Zr,Nb}) \rightarrow \delta\text{UZr}_2$ allotropic reaction and the αU precipitation under different continuous cooling conditions (between 4-120 °C/min).

Considering the thermal evolution experienced by the U-base alloy during the fabrication process of a monolithic-type miniplate U(Zr,Nb)/Zry-4, the concentration domain in which the $\gamma\text{U}(\text{Zr,Nb})$ phase could be retained is around 6-7 wt% Nb. As a consequence, the U-33 wt% Zr-6 wt% Nb alloy has been selected as meat for the fabrication of monolithic-type miniplate U(Zr,Nb)/Zry-4 as second stage of this project.

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