Chapter 3

# Isothermal Phase Transformation of U-Zr-Nb Alloys for Advanced Nuclear Fuels

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## 1. Introduction

Significance in the study of ternary alloys in the U-Zr-Nb system is due to the scenario for their use as nuclear fuel not only for research and test reactors, but also for advanced high flux, power and fast reactors. The importance of this family of alloys is mainly due to the high thermal conductivity, ease of fabrication and good compatibility with the fuel cladding, besides the fact it has high density. Evidently, knowing the basic thermodynamic and diffusion characteristics of this system makes it considerably easier to solve many important problems including the optimization of the fuel composition and their stabilized phase under in-pile conditions. An especially important role in solving such problems is based in the isothermal phase transformation characteristics, which clearly establish the behavior of ternary systems.

The uranium, with a melting point of approximately 1132 °C has three allotropic phases: (i) $\gamma$, high temperature phase with body centered cubic structure, stable between 1132° and 775 °C (ii) $\beta$ phase stable between 775 °C and 668 °C, complex tetragonal structure with 30 atoms per unit cell and (iii) $\alpha$ phase, present at temperatures below 668 °C with orthorhombic structure [1].

For the particular case of U-Zr-Nb system, the introduction of elements zirconium and niobium can result in several structures such as (i) cubic $\gamma^s$ phase, obtained by quenching in water from the high temperature $\gamma$ phase, (ii) tetragonal $\gamma^0$ produced by the aging of $\gamma^s$, (iii) tetragonal $\alpha'$, phase transition of $\alpha$ and (iv) the monoclinic $\alpha''$, transition of $\alpha$ precipitated by aging $\gamma^0$ [2-5].

Since both physical and chemical properties of the nuclear fuel are intimately associated with the current phase, it is impossible to ensure the stability of metallic nuclear fuel without the knowledge of the kinetics of phase transformation in a broad temperature range. For the phase transformations in isothermal conditions, the kinetic behavior of the phase transformation curves is obtained by Time–Temperature-Transformation diagram, known as TTT diagram.

For system U-Zr-Nb, the alloy of composition U-2.5Zr-7.5Nb is the one that has received the largest volume of study in recent decades and different TTT diagrams have been determined as can be seen in Figure 1.

A first investigation in order to determine the TTT diagram for the alloy U-2.5Zr-7.5Nb was performed by Peterson [3] as shown in Figure 1a. This author determined the TTT curves using two main techniques: (i) Rockwell hardness C and (ii) electrical resistivity.

Later, in 1969, Dean [2] made some progress in determining the TTT diagram (Figure 1b) using metallographic techniques and the Vickers hardness tests for phases quantification. Some years later, in an independent and simultaneous way Giraud-Heraud [4] (Figure 1c) and Karnowsky [5] (Figure 1d) determined other two diagrams. The Giraud-Heraud curves, unlike Karnowsky curves, appear to be an improvement of the diagram obtained by Dean as observed on the curves of each diagram.

Besides the determination of the TTT curves, Karnowsky also studied the kinetics of phase transformations based on the equation of Johnson–Mehl–Avrami–Kolmogorov (JMAK) [6] and determined the equation coefficients. An investigation of the kinetics of phase transformation based on equation JMAK was also performed in 1974 by Vandermeer [7] whose TTT curves is shown in Figure 1e. This author introduced an innovation adding a fourth stage in the time-temperature-transformation diagram.

The state of the art on the development of TTT diagrams of U-2.5Zr-7.5Nb alloy still has many gaps and therefore it is necessary to continue parametric investigations and quantifications more precisely of the points near of the TTT diagrams curves. There is already a reasonable consensus on the identification of the phases present in the upper region of the TTT curves for this alloy in the region of temperature between about 400° and 650 °C. But in the range of temperature from about 100° to 400 °C, there are disagreements on the identification and characterization by metallographic technique with regard to the presence of phase $\alpha$''(monoclinic). As reported in the literature, these disagreements may be due to experimental difficulties to reveal their bainitic microstructure.

The alloys of U-Zr-Nb with monoclinic structure $\alpha$" are important to be a stable phase, with excellent mechanical properties and corrosion resistance. Thus, it becomes essential to use during operation of the reactor power in the region of 300 °C. It is observed that for other U-Zr-Nb alloy compositions, unlike U-2.5Zr-7.5Nb, there is an absence of information for construction of the TTT diagrams.

This chapter covers a description of the methodology employed in the study of phase transformation in uranium based alloys with special reference to transformation kinetics under isothermal conditions of system U-Zr-Nb. The determination of the TTT curves in-

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cludes in general, the following stages: (i) uranium alloys obtention, (ii) isothermal treatments, (iii) microstructural and phase characterization and (iv) construction of the time-temperature-transformation curve and study of the kinetics of isothermal phase transformation.

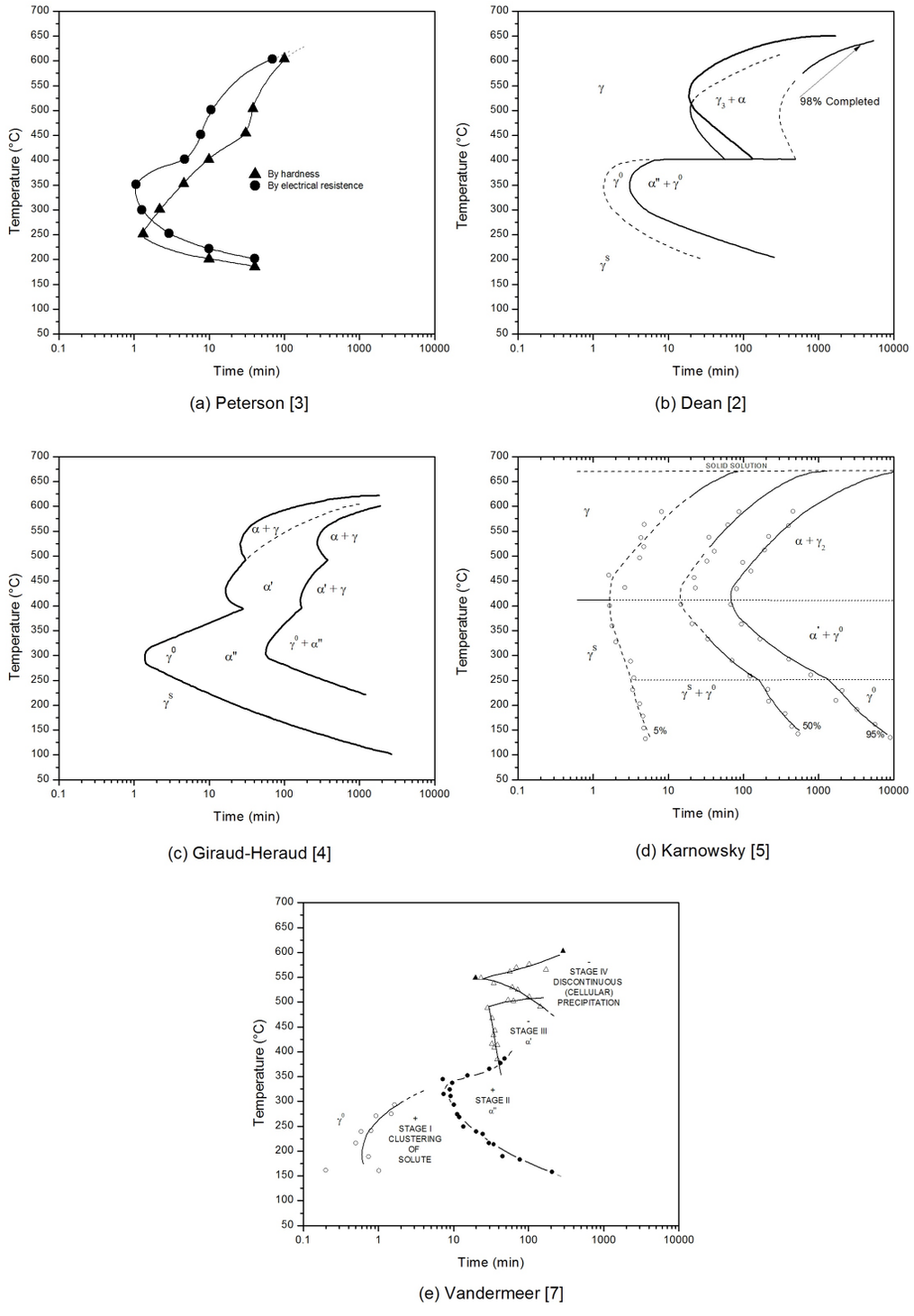


**Figure 1.** TTT diagram of the system U-2.5Zr-7.5Nb obtained by different authors.

## 2. Uranium alloys obtention

The determination of time-temperature-transformation curves for the U-Zr-Nb system requires alloys with chemical and microstructural homogeneity. Several equipments and methodologies can be employed to achieve this goal. In this item we covered a standard strategy employing equipments that are common to many laboratories. Bellow it is presented a simplified methodology and the main challenges to obtain the uranium alloys samples.

### 2.1. Melting of uranium alloys

For uranium alloys obtention it was employed a vacuum induction melting (VIM) furnace. The constituents uranium and alloy elements are melting in a graphite crucible under vacuum up to $1.33x10^{-3}$ Pa at temperatures of about 1500 °C. The time of charge effervescent must be controlled to minimize the carbon contamination and to homogenize the melting charge. The formation of carbides in the alloy structure occurs even in minimal levels of carbon contamination which does not interfere in the TTT diagrams.

After the homogenization, the charge is poured into mold and the casting ingot is cooled at room temperature. The mold can be made of copper or graphite in cylindrical or rectangular shapes.

### 2.2. Alloy homogenization

The alloy homogenization is realized through thermomechanical followed by stress-relieving heat treatments whose goal is to break the alloy cast structure obtaining a homogeneous structure with uniform distribution of chemical elements and equiaxed grains. The stress-relieving heat treatment is conducted at high vacuum ($1.33x10^{-3}$ Pa) and high temperature about 1000 °C and a plateau of approximately 25h.

Among the several hot forming processes employed for breaking the cast structure, the rolling process is undoubtedly the most widely used not only for the uranium-based alloys but for most metals. In this process, breaking of the cast structure is achieved by thickness reduction (about 40%) of the ingot in steps of hot rolling. For these thermomechanical treatments, there are important recommendations in each step as: (i) to avoid excessive oxidation of the ingot using an inert atmosphere and (ii) to prevent warping of the ingot.

## 3. Isothermal treatments

Isothermal treatments in order to determine the transformation-temperature-time curves are schematically shown in Figure 2. These treatments contain several steps in the following sequence named segment A-B, segment B-C, segment C-D and segment E-F.

In the segment A-B, the sample must be heated to the high temperature phase that is the stable phase for the composition of interest and the heating rate in this case is not relevant.

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For the U-Zr-Nb system, the stable phase at high temperatures is the γ phase that. For the composition U-2.5Zr-7.5Nb in weight percent, for example, the temperature of 800 °C corresponds to the stable γ phase. Segment B-C is the region of constant temperature which extension is the time required to complete the stabilization of the high temperature phase. For U-2.5Zr-7.5Nb alloy, the high temperature is the stable γ phase and segment B-C corresponds to about 1 hour.

After segment B-C, the samples follow different conditions according to the isothermal temperature of interest. This temperature is always lower to the segment B-C. Thus, the decreasing in the temperature is performed quickly (down-quenching) until that the chosen temperature T1 is reached, avoiding phase transformations. The quickly down-quenching is especially critical for the isotherms in the knee region of low temperatures (about 300 °C) where the onset of phase transformation occurs in tens of seconds. Usually, this down-quenching is achieved by dipping the sample into metal bath as for example a tin bath or a mixture of lead-tin-bismuth.

Upon reaching the temperature T1, the sample remains in the isotherm for a time t1 and is rapidly cooled in order to retain the formed structure (segment E-F). The same procedure is adopted for the chosen times as t2, t3, t4, t5 etc. (Figure 2). Due to the nature of the process of isothermal transformation, the times are given on a logarithmic scale.

The retention structure is realized rapidly by transferring the sample to the room temperature in water or liquid nitrogen. After this quenching, the structure of the sample is characterized to determine the current transformed phase fractions. Thus it is possible to investigate the phase transformations in function of times of isothermal treatments. For practical purposes, the phases are determined in the range from 5 to 95% of transformation. The TTT diagrams exhibit C-curves or unusual double-C curves.

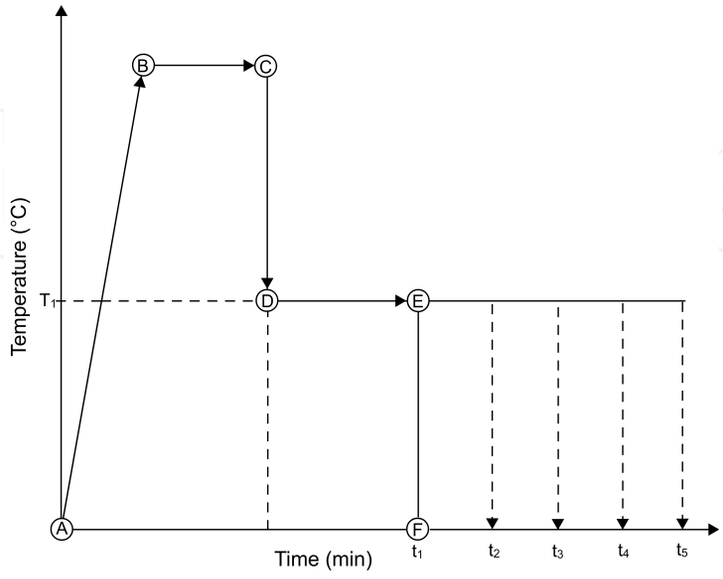


**Figure 2.** Schematic configuration of the isothermal treatments.

### 3.1. Installation for isothermal treatments developed at CDTN

We developed in the Researh Institute - CDTN (Centro de Desenvolvimento da Tecnologia Nuclear) an innovative isothermal treatments installation with precise monitoring of all steps involved in the procedures adopted to realize the isothermal treatments as described above.

This isothermal treatments installation comprises of three main parts: (i) high temperature zone, (ii) isothermal treatment zone and (iii) cooling zone as shown in Figure 3. The different zones are interconnected by a steel tube that is cooled between the zones by a water cooling system.

The tube ends are coupled to the left a pressure gauge, a vacuum pump and a gas outlet valve and to the right a gas control valve and a system that allows movement of the samples whose function is to transfer the sample between the different zones. This system has a haste connected to the sample crucible whose displacement is controlled by software what allows a rapid and accurate haste shift, so that the sample can be moved from the high temperature zone to cooling zone in in a few seconds. Also to the right there is a tempering container for quenching in water or oil, or liquid nitrogen.

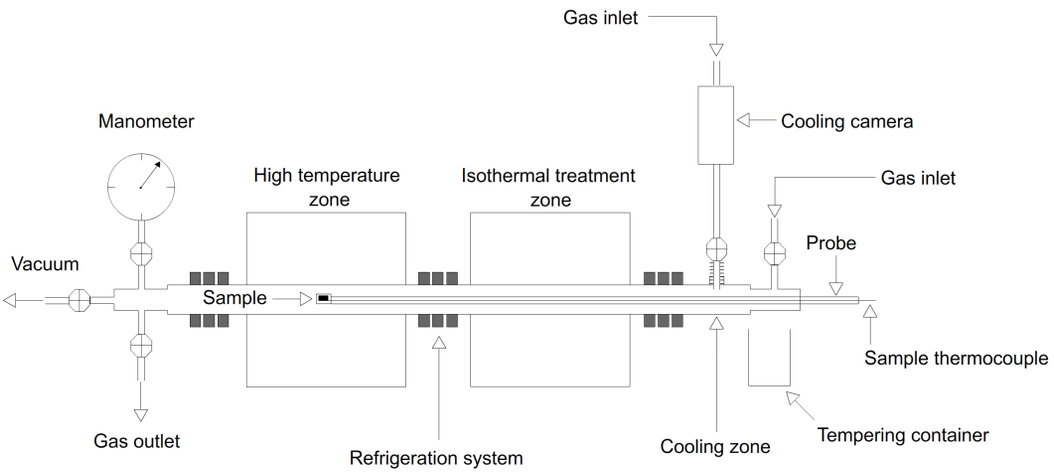


**Figure 3.** Isothermal treatments installation for phase transformations.

In order to perform the experiments, the tube is evacuated to about $1.33 \times 10^4$ Pa and in the following inert gas is injected. This procedure is carried out five times to ensure the removal of oxygen inside of the tube. Next, the high temperature zone is activated reaching the temperature of 800 °C, at a rate of 10 °C/min. At this time, the isothermal treatment zone is heated up to the desired isothermal treatment temperature. After reaching the temperature of 800 °C, the sample is shifted to the high temperature zone. The sample remains in this region for an hour in order to stabilize the high temperature $\gamma$ phase. In the following, the sample is cooled until isothermal temperature and time programed. Finally, once reached the time of isothermal treatment the sample is quenched in water.

This innovative isothermal treatments installation eliminates the quenching in tin bath or mixture of lead-tin-bismuth between the zones of high temperature and isothermal temperature. Furthermore, this automated installation ensures a great reproducibility of the experiments.

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## 4. Microstructural and phase characterizations

A variety of different techniques can be used for microstructural and phase characterizations as metallography, Vickers microhardness, X-ray diffraction, dilatometry, electrical resistivity, thermal measurement techniques, magnetic permeability among others. Generally, more than one technique can be used in a complementary way.

Below are described some techniques used to obtain the TTT curves for the system U-Zr-Nb showing some limitations and difficulties as also the samples preparation applied on each technique.

The most common techniques used to determine the TTT curves that are available in most laboratories are: (i) optical microscopy, (ii) scanning electron microscopy (SEM) coupled with energy dispersive spectroscopy X-ray (EDS), (iii) Vickers microhardness (HV), (iv) X-ray diffraction (DRX) associated with the Rietveld method.

### 4.1. Sample preparation

In the first time, the samples should be taken randomly and prepared by conventional metallographic techniques as cutting, mounting, grinding and mechanical and electrolytic polishing until to obtain a specular surface.

Optical microscopy is employed to observe mainly precipitates, phases and grains. To reveal the grains it is utilized electrolytic etching with a common solution 10wt% of oxalic acid in water. The revelation of the grains is a difficult task for the U-Zr-Nb alloys. The major difficulty is the revelation of phases formed on the isotherms for low-temperature region where microstructural characteristics of phase transformation are not clear. In order to overcome this problem it is necessary an extensive study aimed at determining optimized solutions of chemical attack for this alloys. For the 600 °C isotherm, for example, the microstructure is easily revealed, and there is no problem in identifying the progress of the transformation. The electrolytic attack and their solutions are well known in these conditions.

For X-ray diffraction analysis, the sample preparation is identical to metallographic procedure presented above but without electrolytic etching. Due to the high density of the uranium in these alloys, analysis by X-ray diffraction is restricted only to some atomic layers. Thus, care must be taken in metallographic preparation to avoid mechanical deformations induced on the sample surface. These deformations are manifested as broadening of the Bragg reflections making it difficult to analysis phases mainly because the uranium metastable phases have peaks very close together.

The sample preparation is identical to metallographic procedure above for the techniques as Vickers microhardness, scanning electron microscopy and energy dispersive spectroscopy X-ray.

### 4.2. Optical microscopy

The optical microscopy is one of the techniques used in the construction of TTT diagrams. As mentioned above, good quality of the micrographs is related to an adequate sample surface which is not always easy to obtain for the U-Zr-Nb system, especially for low-temperature isotherms.

Some optical micrographies are presented in Figure 4 as examples of the evolution of the TTT diagrams for U-3Zr-9Nb alloy treated isothermally at 600 °C for times of 100, 1000 and 10,000 min, utilizing the isothermal treatment installation (Figure 3). Similar microstructural characteristics are also observed for the U-2.5Zr-7.5Nb system submitted to the same thermal treatment conditions.

As can be seen in the Figure 4, the phase transformation $\gamma \rightarrow \alpha + \gamma$ occurs during up to 10,000 min. The $\gamma$ phase appears as a single phase at 100 minutes (Figure 4a) and in Figure 4b there is clearly distinct regions, i.e., untransformed $\gamma$ phase and a perlitic phase ($\alpha + \gamma$). Already at 10,000 minutes (Figure 4c) a perlitic structure is present in the entire sample. Thus, the perlitic structure grows gradually over the whole sample.

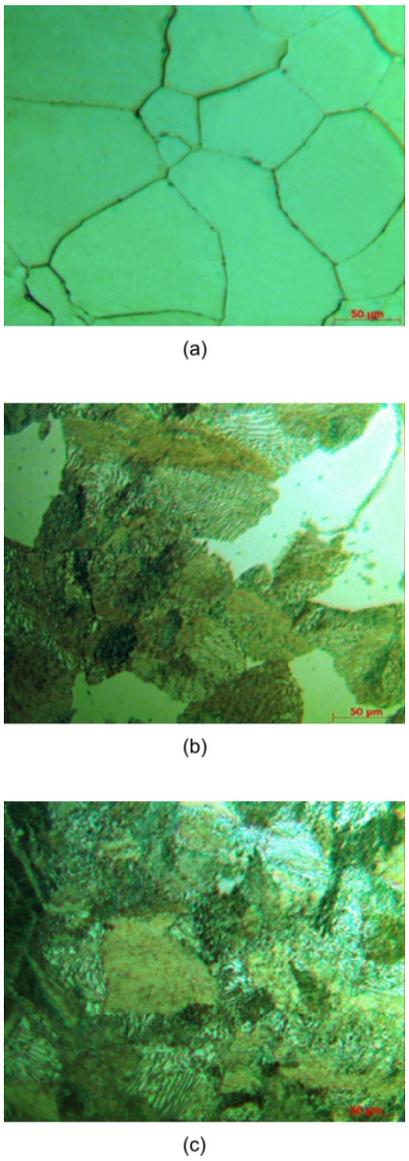


**Figure 4.** Optical micrographies of U-3Zr-9Nb alloy isothermally transformed at 600 °C for (a) 100 min, (b) 1000 min and (c) 10,000 min.

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The fraction of transformed phases for construction of the TTT diagram can be performed through software image analyzer. The Quantikov® software [8], which was developed at CDTN is utilized for quantification of the phases of U-Zr-Nb alloys and includes modules for digital image processing, geometric measurements, graphics and hypertexts.

### 4.3. Scanning electron microscopy and X-ray energy dispersive spectrometer

Scanning electron microscopy coupled with energy dispersive spectroscopy of X-rays is another technique employed to construct the TTT diagrams for the U-Zr-Nb system. A wide range of high resolution and depth of field of the SEM analysis allows to obtain images with three-dimensional appearance showing microstructural details not revealed in the optical microscope. Figure 5 shows some SEM micrographies for U-3Zr-9Nb alloy obtained from studies in U-Zr-Nb alloys in our laboratory. From this figure, the lamellae can be observed in more detail compared with the optical micrographies. Figure 5b emphasizes the lamellar structure during the isothermal treatment at 10,000 minutes.

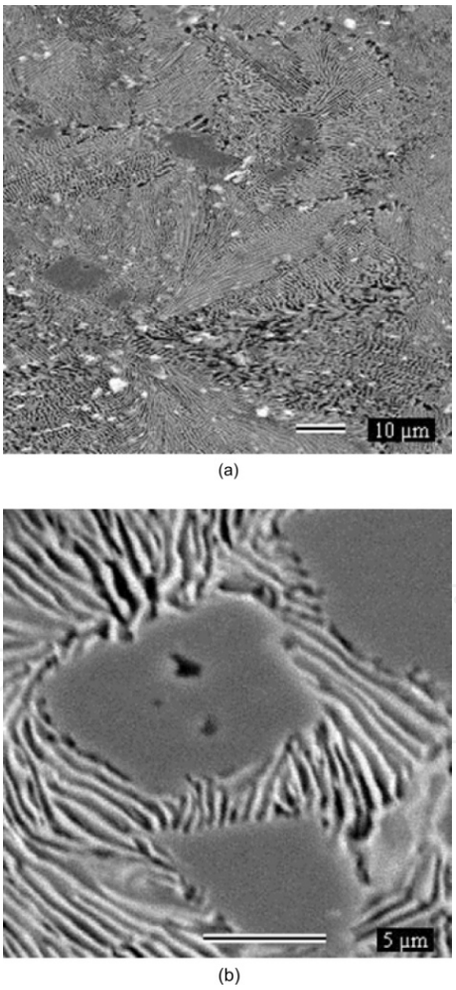


**Figure 5.** SEM micrographies of U-3Zr-9Nb alloy isothermally transformed at 600 °C for 10,000 min with different magnifications.

The chemical microanalysis of each element in the alloys are simultaneously detected by the EDS technique. This technique has advantage to allow chemical analysis on a very small region on the samples. This chemical analysis can be performed by two ways: (i) stationary, when the electron probe remains in a given position until complete X-ray events are recorded by the detector and (ii) scanning, when the electron probe moves over a line on the samples covering a determined length [9]. For the system U-Zr-Nb both modes of analysis are important. The stationary mode may be employed, for example, for microanalysis of precipitates and different chemical structures observed in the sample. The scanning mode is particularly useful to verify the concentration of a particular element across different regions.

An example of EDS analysis is shown in Figure 6 where the concentration of the elements uranium, niobium and zirconium are monitored along a given line length. On the left upper side of the figure, the micrograph of the sample shows, the presence of a precipitate (dark region) embedded in the matrix (light region). The drawn arrow passes through the matrix and precipitate indicating the direction path of electron probe. It is observed in this graph a sharp change in elements composition in the precipitate region showing the predominance of the elements niobium and zirconium.

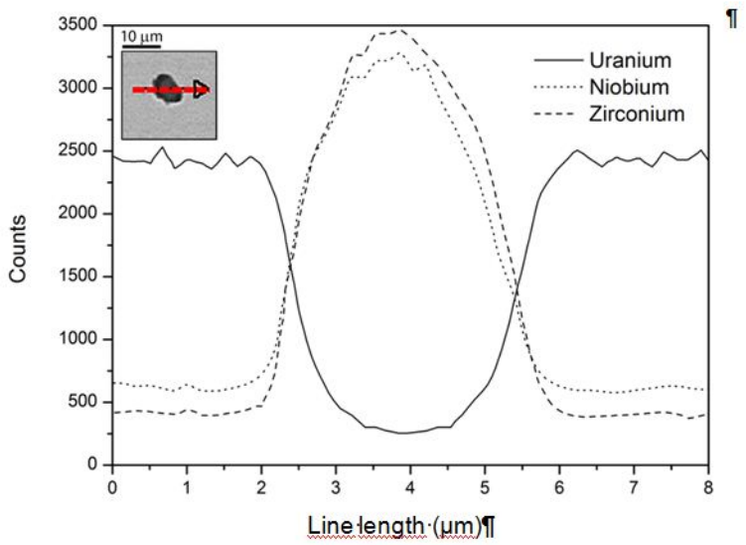


**Figure 6.** EDS chemical microanalysis on the U-2.5Zr-7.5Nb alloy.

### 4.4. Vickers microhardness

The Vickers microhardness is a technique widely used to study phase transformations in alloys under isothermal conditions especially due to their sensitivity to distinguish different phases. For the U-Zr-Nb system , Vickers microhardness has a particular importance for assessing the evolution of the phase transformations especially for a low temperature isotherm region. As mentioned earlier, this region is difficult to be analyzed by microscopy due to the difficulty in revealing the structure.

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Vickers microhardness results, as shown in Figure 7, present an increase in the microhardness from the high-temperature isothermal phase transformations to regions of low temperature (about 300 °C). In this region of low temperature, the microhardness continuously increases with aging time. These results have similar behavior with Dean results [2]. But, in the high temperature region, approximately 600 °C, Dean microhardness results show an abrupt increasing conflicting very much from our results. Faced the differences mentioned it is important to mention that the microhardness results are very scarce in the literature for the U-Zr-Nb alloys what it makes necessary to investigate the microhardness in these alloy in a systematic way covering this shortcoming.

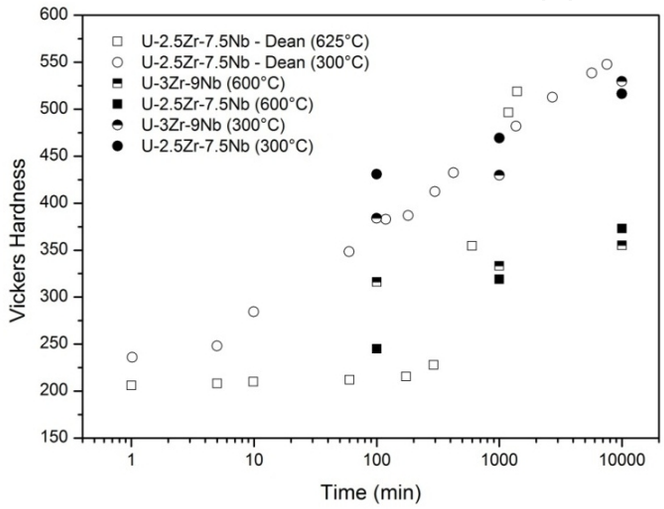


**Figure 7.** Vickers microhardness results for U-Zr-Nb alloys.

### 4.5. X-ray Diffraction

The X-ray diffraction is the main technique used to study the phase transformations in materials. A comprehensive study of X-ray diffraction for the U-2.5Zr-7.5Nb alloy was conducted by Yakel in 1976 [10], where the main metastable phases were identified. Uranium based alloys have a high absorption coefficient for X-ray which limits diffraction analysis in the surface layers of samples making it dependent on the surface preparation as discussed above. Additionally, patterns of X-ray diffraction peaks exhibit excessive enlargement which also contributes to increase the difficulties of the analysis particularly where the proximity of Bragg reflections are very close.

In order to overcome these problems, the Rietveld method emerges as a powerful technique for analyzing the X-ray diffraction data. The Rietveld method works to reduce the difference between the observed experimental diffraction pattern and the calculated diffraction pattern from a known structural model. In other words, the calculated diffraction pattern is modified by adjusting specific parameters to be as close as possible to the experimental pattern. This allows extract information of material structure, e.g., adjusted lattice parameter and quantitative analysis of phases, making the method a relevant tool in the studies of the

phase transformation in the system U-Zr-Nb. A complete description of the mathematical involved in the Rietveld analysis can be obtained in the reference [11].

Figure 8 shows a typical Rietveld analysis for the U-2.5Zr-7.5Nb and U-3Zr-9Nb alloys. Both alloys were subjected to isothermal treatment at 600 °C for 10,000 minutes. As can be seen in the patterns of Figure 8, only perlitic structure are present indicating the occurrence of $\gamma$ phase transformation to $\alpha + \gamma$. In this analysis, the red points corresponds the experimental diffractograms, the green line represents the calculated diffractogram and the magenta line is the difference between the observed and calculated patterns. Black and red marks correspond to Bragg reflections of $\gamma$ and $\alpha$ phases, respectively. Through the intensity of the main peaks, it can be inferred that the $\alpha$ phase transformation is highest in the alloy with a lower content of alloying elements, i.e., U-2.5Zr-7.5Nb alloy. The accurate quantification of the percentage of transformed phase and the evaluation of the lattice parameter of the structures formed are the major contribution of the Rietveld method to study the isothermal transformation system in U-Zr-Nb.

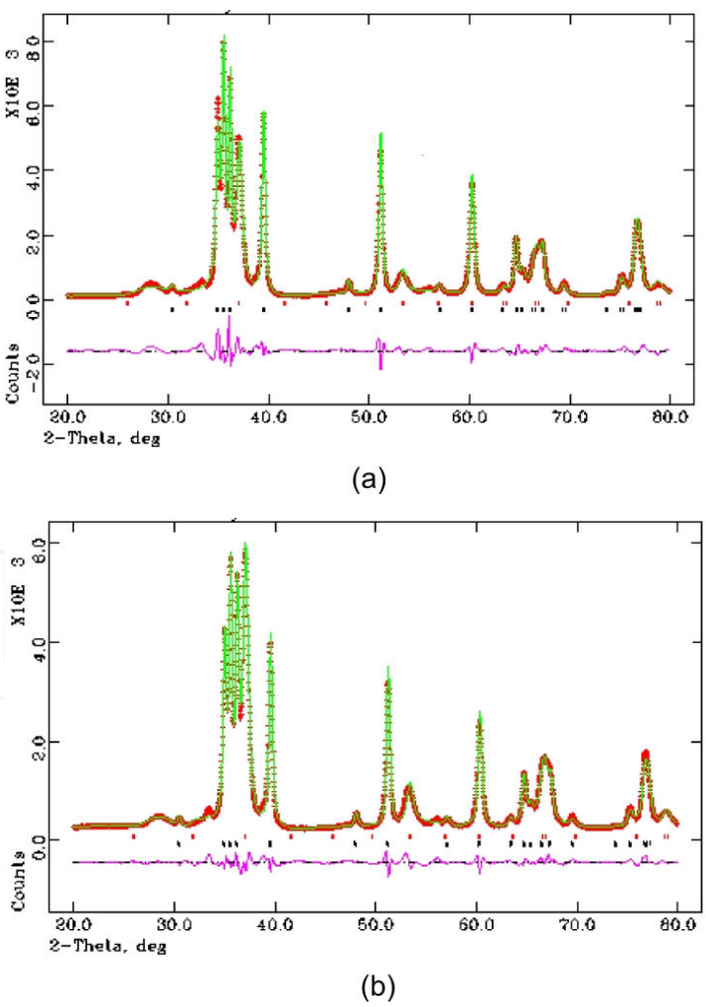


**Figure 8.** Rietveld refinements of the alloys aged 24 hours at 600 °C: (a) U-2.5Zr-7.5Nb, (b) U-3Zr-9Nb.

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## 5. Construction of the time-temperature-transformation curve and study the kinetics of phase transformation

We reported that isothermal treatments in function of time are needed to determine the transformation-temperature-time curves being the time given on a logarithmic scale due to the nature of the process of isothermal transformation. It is also reported the techniques utilized for determining the percentage of phase transformed for construction of TTT diagrams. Thus, the TTT diagrams are constructed through isothermal treatments accomplished by phase characterization.

For kinetics studies of phase transformations, it is necessary to construct curves, usually referred to S-curves. In these curves, the transformed phase fractions are given as a function of time of transformation and important informations about the isothermal as nucleation time, start--transformation time, half-transformation time half and end-transformation time.

The S-curves for the U-2.5Zr-7.5Nb alloy adapted from Vandermeer [7] and Karnowsky [5] at different temperatures are shown in Figure 9 and it can be noted that the temperature change the shape of the transformation S-curves as a result of its influence on the kinetics of phase transformation.

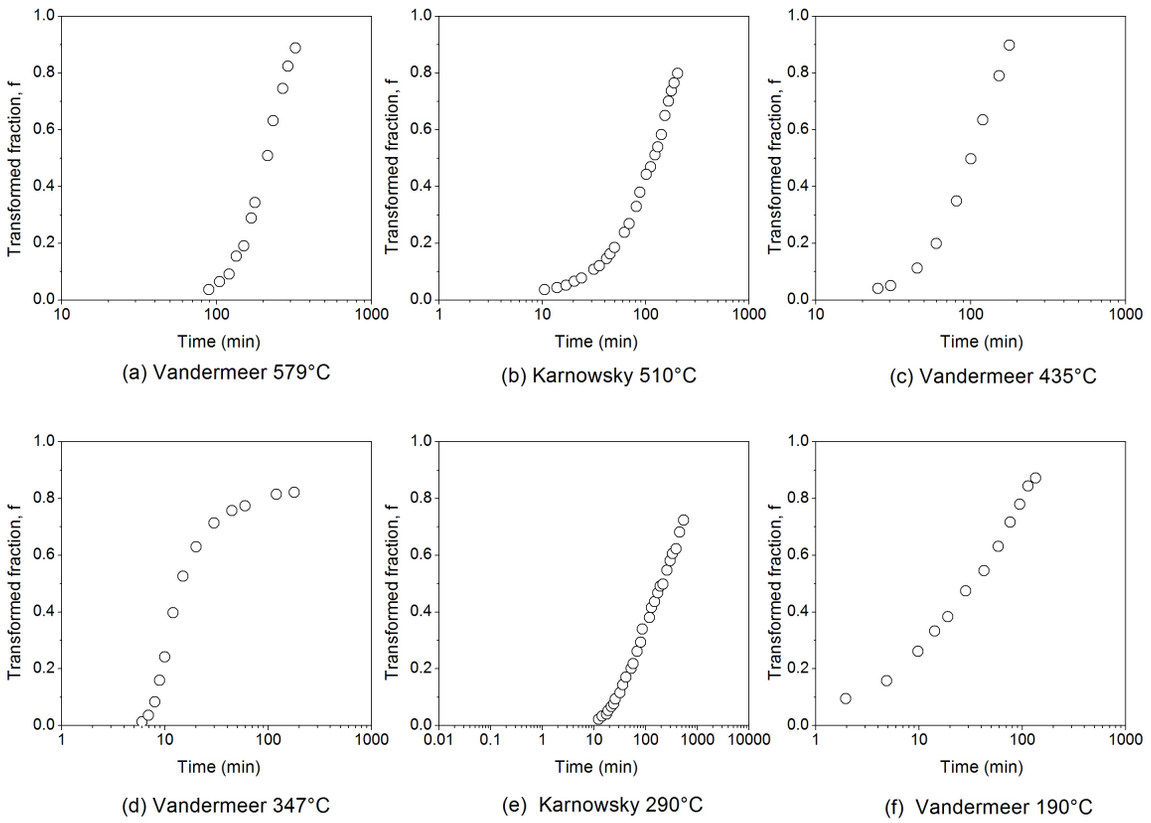


**Figure 9.** S-curves for the U-2.5Zr-7.5Nb alloy. (a), (c) (d) and (f) adapted from [7]; (b) and (e) from [5].

The determination of S-curves is generally accompanied by a study of the kinetics of phase transformation under isothermal conditions through the phenomenological model described by Johnson-Mehl-Avrami-Kolmogorov and represented by the following equation:

$$f = 1 - exp(-kt^n) \quad (1)$$

where f is the fraction transformed, t is the aging time and k and n are constants. The value of n constant allows to perform a series of inferences on the mechanism of phase transformation under isothermal conditions. The constants n and k can be obtained by linearization of this equation where n is the slope of the curve and k is the interception of y axis. Figure 10 shows the linearized curves of the isothermal temperatures of 579° C and 510 ° C which S-curves are shown in Figures 9a and 9b. Through the determined n values, the mechanisms of phase transformations can be inferred in Christian work [6].

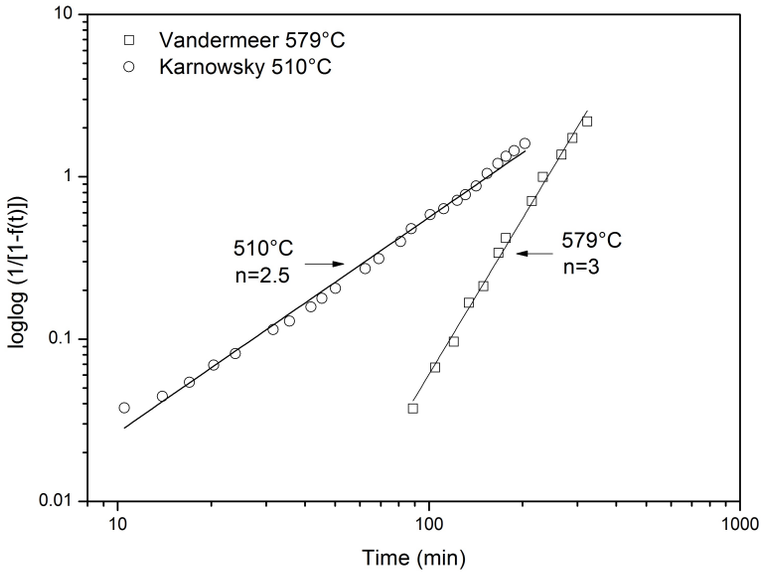


**Figure 10.** Linearized curves of the isothermal temperatures of 579 °C and 510 °C (adapted from [5] and [7]).

If the kinetics of phase transformation is governed by only one mechanism, the curve loglog (1/(1-f)) vs. log t shows a linear behavior as that shown in Figure 10 for the U-2.5Zr-7.5Nb alloy for the isotherms of high temperature. However, in the case of low isothermal temperature the U-2.5Zr-7.5Nb alloy does not exhibit a curve of linear behavior. Instead of the curves present two regions with different slopes indicating the occurrence of two mechanisms governing the kinetics of isothermal phase transformations. The complete elucidation of the mechanisms present in the phase transformations for U-2.5Zr-7.5Nb alloy is still at a preliminary stage and needs to be extensively investigated.

The construction of the TTT diagrams can be accomplished by the transfer of the S- curves as shown schematically in Figure 11.

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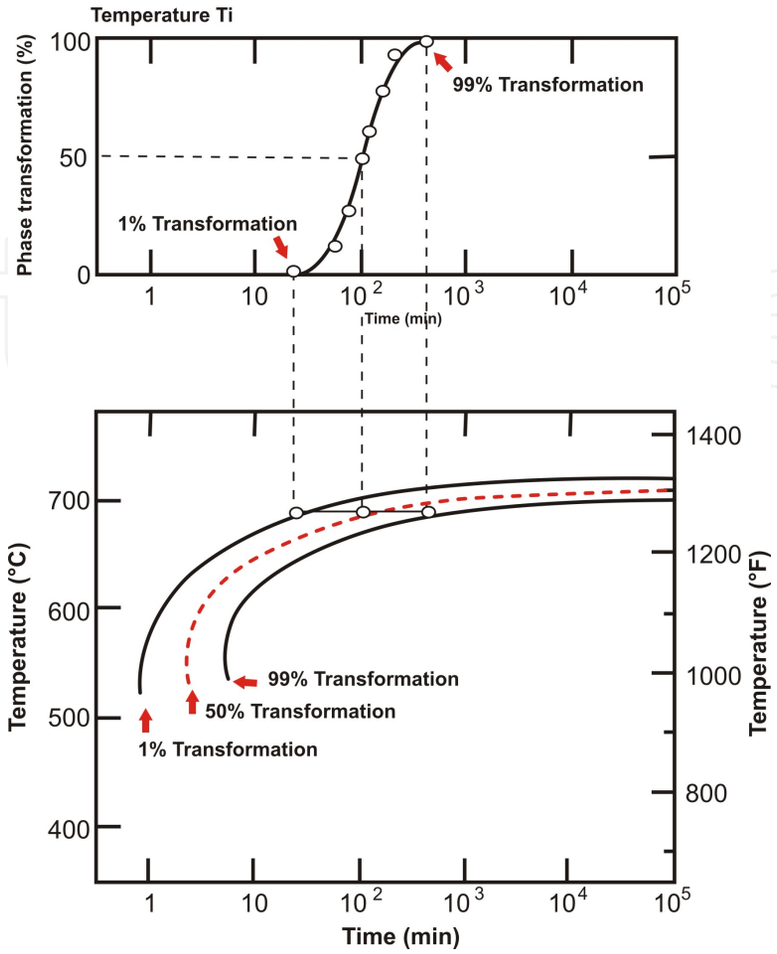


**Figure 11.** Scheme of construction of the TTT diagrams from S- curves (adapted from [12]).

## 6. Conclusions

This Chapter presents the state of the art of TTT diagrams of the U-2.5Zr-7.5Nb alloy as well as the development about this subject in the CDTN. It was observed that the study of the phase transformations and TTT diagrams is more focused on U2.5Zr7.5Nb alloy and even thus there are many disagreements in the phase characterization mainly in the phases present in the isothermal regions of lower temperatures. Moreover, a huge shortcoming exists in the literature about the development of TTT diagrams in other compositions of the U-Zr-Nb system.

In this context, orientations to study the isothermal phase transformations and to develop the TTT diagrams for the U-Zr-Nb system were presented. Furthermore, the methods and techniques employed as well as the care involved in each experimental step are also shown.

Summarizing the main informations in this Chapter are listed below:

- It was shown the main techniques utilized in the development of TTT diagrams for the U-Zr-Nb system;
- Experimental results carried out at CDTN for isotherms at 300 °C and 600 °C were presented;
- Finally, it was described an innovative installation for isothermal treatments developed at CDTN. This allows that the study of isothermal phase transformations in the U-Zr-Nb system will be performed in a more accurate way by monitoring of the involved stages in the procedures adopted. Thus, further diagrams will be constructed with isotherms in the temperature range between about 100 ° to 650 ° C for alloys of U-2.5Zr-7.5Nb and U-3Zr-9Nb.

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