

# INVESTIGATION OF MECHANICAL PROPERTIES OF RESINTERED UO<sub>2</sub> PELLETS BY MICRO HARDNESS METHOD

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## YENİDEN SİNERLENEN UO<sub>2</sub> PELETLERİNİN MEKANİK ÖZELLİKLERİNİN MİKRO SERTLİK METODUYLA İNCELENMESİ

### Abstract:

Thermal stability test (re-sintering test) of the nuclear fuel is one of the most important characterization steps before loading to the reactor. The purpose of this study is investigation of mechanical properties of re-sintered UO<sub>2</sub> pellets using micro hardness test method. UO<sub>2</sub> fuel pellets were produced by powder metallurgical route. UO<sub>2</sub> pellets sintered for 4 hours at Ar + 5% H<sub>2</sub> atmosphere were subjected to re-sintering at 1700 °C for 24 hours in Ar + 5% H<sub>2</sub> atmosphere for thermal stability test. Re-sintering effects on the mechanical properties and microstructure of UO<sub>2</sub> pellets were investigated.

### Özet:

Termal kararlılık testi yakıtların reaktöre yüklenmesinden önce uygulanan en önemli testlerden biridir. Bu çalışmanın amacı yeniden sinterlenen UO<sub>2</sub> peletlerinin mekanik özelliklerinin mikro sertlik yöntemiyle belirlenmesidir. UO<sub>2</sub> peletleri toz metalürjisi yöntemiyle üretilmişlerdir. UO<sub>2</sub> peletleri Ar +% 5 H<sub>2</sub> atmosferinde 1700 °C'de 4 saat süreyle sinterlendikten sonra aynı atmosfer ve sıcaklıkta 24 saat süreyle yeniden sinterlenerek termal kararlılık testine tabi tutulmuştur. Yeniden sinterleme işleminin UO<sub>2</sub> peletlerinin mekanik özelliklerine ve mikro yapısına etkisi incelenmiştir.

**Keywords:** UO<sub>2</sub>, re-sintering, thermal stability test, micro hardness, mechanical properties, microstructure

**Anahtar kelimeler:** UO<sub>2</sub>, yeniden sinterleme, termal kararlılık testi, mikro sertlik, mekanik özellikler, mikro yapı

### 1. Introduction

Nuclear fuels have specific quality standards. Thermal stability test is one of the quality tests applied for UO<sub>2</sub> nuclear fuel pellets (bsi-DPC:12/30254328DC, 2012). Thermal stability of nuclear fuel is very important for structural integrity of nuclear fuel under reactor operating conditions. Fission gases formed under reactor operating conditions are accumulated in the pores of nuclear fuels. Meanwhile, new gas bubbles and fission products are developed in the nuclear fuel pellets. All these effect leads to structural defects and dimensional changes in the sintered fuel.

One of the most important problems in the safe and reliable operation of power reactors is that the fuel performance tests could not be performed completely. Since the in-pile thermal stability test is quite long, complicated and expensive, it is not widely used. On the other hand, the out-of-pile re-sintering / thermal stability test does not precisely predict fuel behavior. Nevertheless, thermal stability test is generally used to predict the behavior of fuel pellets in

the reactor (Basov, 2009). In the thermal stability test, the pre-test densities of the pellets are determined, then the pellets are sintered at 1700 °C, UO<sub>2</sub> pellets re-sintered in the same conditions for 24 hours. Then the re-sintering densities were recalculated. The density changes, microstructure, dimensional changes and mass changes were determined for thermal stability parameters of fuel.

Grain size and pore size are important parameters for the thermal stability. The decreasing of the pore size leads to the thermal stability decrease. At the same time, irradiation stability decreases. The pore distribution below 5 µm should be minimal to ensure the irradiation stability of the pellets, (Kim, Kim, Joung, Lee, & Sohn, 2002,). Pore size distribution and homogeneity of pore distribution are important for thermal stability too.

The high temperature sintering under H<sub>2</sub> atmosphere of UO<sub>2</sub> pellets reduces the porosity. The dimensional change of the porosity is important during irradiation since the pores will shrink due to the fuel temperature and therefore the pore size distribution will be changed. The porosity affects the dimensional stability of the pellets (Paraschiv, Paraschiv, & Grecu, 2002). The changing ratio of diameter, height and density of the pellets are important parameters for the thermal stability test (Basov, 2009).

In this study, the density, microstructure and micro hardness of pellets were investigated before and after the re-sintering process. The results of the measurements of re-sintered UO<sub>2</sub> pellets were compared with some earlier studies. Additionally, “micro hardness measurements” was performed for predicting the mechanical behavior of re-sintered pellets. Note that, there could not found any study in the literature that including micro hardness testing for resintered UO<sub>2</sub> pellet. The results obtained from the measurements were remarkable.

## **2. Materials and Methods**

### **2.1 Specimen preparation**

In this study, UO<sub>2</sub> pellets were produced by powder metallurgical route. Nuclear fuel powders produced by ex-ADU (Ammonium diuranate) route . The UO<sub>2</sub> powders were mixed with zinc stearate as a binder and lubricant (0.2 wt. %) and mixed for 12 hours in a drum mixer. The powder was pressed in a 15 mm cylindrical die under 300 MPa pressure. The green pellets had 50% theoretical density (%TD). The pellets were sintered at 1700 ° C for 4 hours in Ar+5%H<sub>2</sub> atmosphere, UO<sub>2</sub> pellets re-sintered in the same atmosphere for 24 hours at 1700 °C (heating rate of 10 K/min). The re-sintering density ( $\rho_{rs}$ ) values were determined using water immersion method. The relative density ( $\rho' = \rho / \rho_{TD} * 100$ ) values of UO<sub>2</sub> pellets before and after the re-sintering for thermal stability test were 96.33 and 95.69 TD% respectively. The density changing versus the re-sintering time of UO<sub>2</sub> pellets is given in Figure 1.

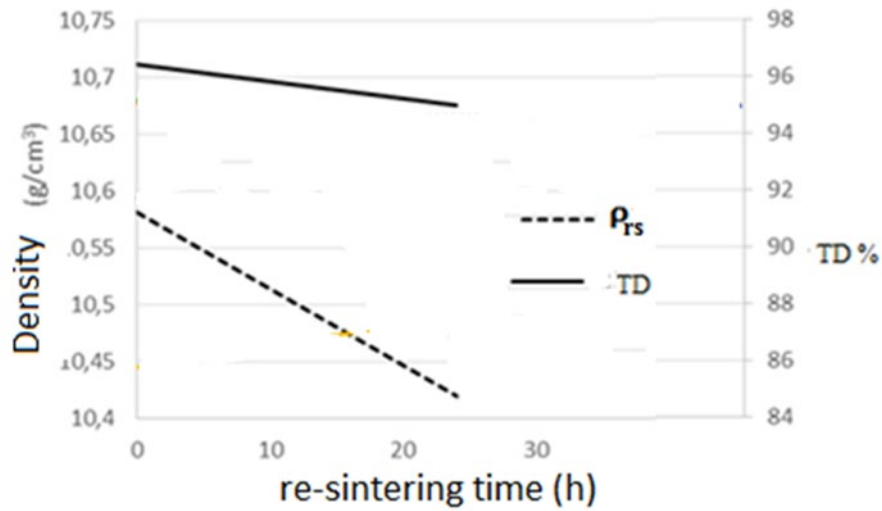


Figure 1: Density change versus re-sinter time in UO<sub>2</sub> pellets

## 2.2. Micro Structural Analyses

Microstructural investigation was carried out by ceramographical method and grain size analysis on the UO<sub>2</sub> pellet surfaces performed by using Analsis-5 program. The microstructure image of the UO<sub>2</sub> pellet after resintering is given in Figure 2 (Scanning Electron Microscope-SEM). The average grain sizes of pellets before and after thermal test is 13 μm and 44 μm respectively.

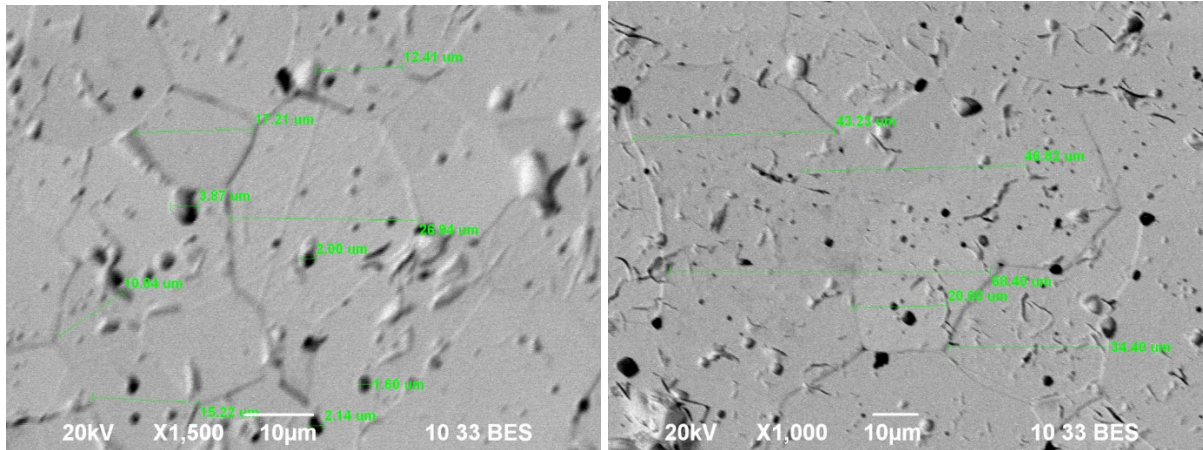


Figure 2: SEM micrographs of sintered and re-sintered UO<sub>2</sub> pellets

## 2.3 Micro Hardness Measurement

Hardness measurement determines the resistance to the plastic deformation of the material. In order to obtain the hardness value, the specially shaped small in is applied to the sample surface and then the hardness value is determined from the size or depth of the groove formed on the sample surface. Brinell, Vickers, Knoop and Rockwell standard test methods are used to establish the relationship between notch size and hardness. There is no single best test or even a common scale to measure the hardness of the sintered materials. Care should be taken to ensure that the tip of the indenter does not hit any pore during measurements. In Vickers method it is easier to adjust the measurement point under the microscope and thus to

avoid measurement on pore areas. Since the pores does not exhibit any hardness, the macro hardness will be lower than the micro hardness values (German, 2007).

Vickers hardness measurement method bases on optical measurement of the indentation of a  $136^\circ$  diamond pyramid tip, which is applied with a certain force on the material selected depending on the material type and thickness. The schematic presentation of the Vickers hardness measurement system is given in Figure 3, where  $F$  is the applied force (kgf);  $d_1$ (mm) and  $d_2$  (mm) are the diagonal dimensions of the indentation, and  $x$ ,  $y$  and  $z$  are the coordinates. The average diagonal dimension ( $d_{avr}$ ) was taken in the calculations.

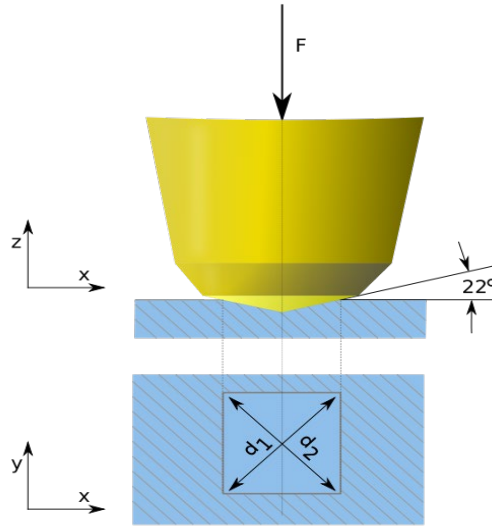


Figure 3: Schematic of Vickers measurement system

Vickers hardness value (HV) is calculated in units of  $\text{kgf} / \text{mm}^2$  by using Equation 1, where  $F$  is the applied force and  $A$  is the unit area. The value of the HV is usually between 0 and 15 GPa.

$$\mathbf{HV} = \frac{F}{A} \approx \frac{1,8544F}{d_{avr}^2} \quad (1)$$

In this study, DURASCAN20 measurement system was used for Vickers experiments. The test was carried out on sintered and re-sintered pellets at different load values ( $0.025 \text{ kgf} \leq F \leq 1 \text{ kgf}$ ) for 10 seconds on polished surfaces. The applied load was perpendicular to the pressing direction ( $\perp$ ). Load-dependent micro hardness change graphs applied before and after the re-sintering of  $\text{UO}_2$  pellets are given in Figure 4. Micro hardness (MH) testing and optical micrographs of  $\text{UO}_2$  pellet are given in Figure 5. The comparison of the micro hardness values of  $\text{UO}_2$  pellets with the literature is given in Table 1.

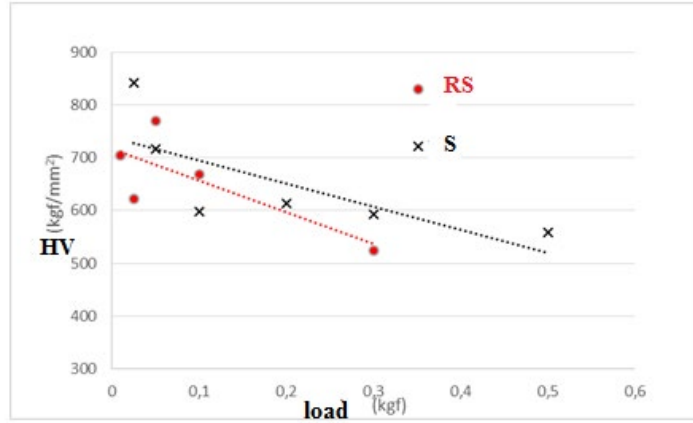


Figure 4: Micro hardness measurement values of sintered and re-sintered UO<sub>2</sub> pellets

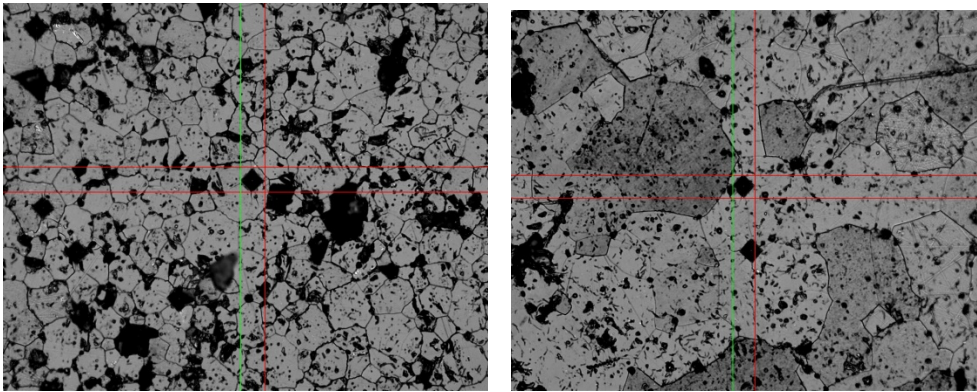


Figure 5: A view from the indentations on the optical micrographs of the UO<sub>2</sub> sintered (left) and re-sintered (right) pellets (x20)

Table 1: Comparison of micro hardness values of UO<sub>2</sub> pellets with literature

Load F 0,3 kgf	HV (kgf/mm <sup>2</sup> )		
	(Artir, Aybers, Akşit, & Akbal, 2006)	Sintered	ReSintered
HV	526	592	525
%TD	97	96	95.7
GS (µm)	10,8	13	44

### 3. Results and Discussion

Thermal treatment of the material changes its mechanical properties. In this study thermal stability tests were applied on sintered UO<sub>2</sub> pellets and then mechanical properties were determined by the micro hardness measurement method. The results are summarized as following.

- The mean theoretical density values before and after the re-sintering were 96.33% and 95.69% respectively.
- These results are similar to some literature studies as shown in Figure 1.
- The average particle sizes before and after the re-sintering are 13 µm and 44 µm respectively.
- Sintered and resintered micro hardness values were determined as 592 and 525 kgf/mm<sup>2</sup> respectively.
- The grain size increased after re-sintering, and the corresponding micro hardness value decreased.

### 4. References

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