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Atomization of UMo Particles under Nitrogen Atmosphere

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Abstract

Generally, the atomization of UMo particles is done under vacuum or argon atmosphere, and the surface modification of these UMo particles is, usually, carried on through a further process. The techniques for surface modification of atomized UMo particles, aimed to control the Fuel/Matrix interaction, involve, in some cases, complex methodologies and often with minor effect due to the limited solubility of third elements in solid UMo alloy. The atomization and surface conditioning, applied in separate stages, may affect the efficiency of powder production process. Then, the main goal of this study is to explore the surface modification of UMo particles in liquid state or during the solidification that follows the centrifugal atomization process. Through the change of atomization atmosphere, could be possible to promote liquid/gas reactions, with a higher solubility of the modifier element in micro drops of UMo alloy, before they become solid particles. This paper presents comparative results of centrifugal atomization of UMo particles, carried out under inert argon and reactive nitrogen atmospheres. Dissolved nitrogen contents, measured by SEM-EDS analyses, reached up to 7.57 wt% at the center of under nitrogen atomized particles, very higher than 0.84 wt% of nitrogen measured at the center of UMo particle atomized under argon. The presence of uranium nitride was partially verified by conventional XRD analysis. Nevertheless, Out-of-Pile interaction test result, reveals decreasing of aluminium contents into UMo particles atomized under nitrogen atmosphere; Just 3.77 wt% of Al was the maximum content detected in the center of these particles, very lower than 29.11 wt% of Al measured inside UMo particles atomized under argon. Finally, it is possible to conclude that the atomization under reactive atmosphere may modify the surface composition and the behavior of UMo fuel particles dispersed in aluminium, for dispersion type nuclear fuel application.

Keywords

Atomization, UMo Particles, Dispersion, Nuclear Fuel

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1. Introduction

The qualification of high density nuclear fuel based on UMo alloy requires, necessarily, solve the fuel/matrix interaction issues. Several articles, which cover in detail that topic, have already been written and published by nuclear fuel experts and also by researchers of the CCHEN's Fuel Development Group [1]. An effective way to control the interdiffusion between aluminum matrix and UMo fuel, is related to the surface coating of the UMo particles, so as to form diffusion barriers, which can help to avoid or delay the interaction layer occurrence, and the consequent formation of compounds with poor in-reactor performances, as $U_6Mo_4Al_{43}$ and/or UAl_4 [2].

According to the available literature, several methodologies have been developed for surface coating of UMo particles, some of them based on solid-solid reactions, as the pack cementation technique to form silicon-rich surface layers [3], others based on solid-gas reactions, such as thermal treatments under nitrogen atmosphere to form uranium nitride layers [4], and solid-plasma reactions, such as PVD or CVD applied to the formation of Zr or ZrN layer [5]. In general, all these methodologies are carried out with UMo fuel particles in solid state, which limits the solubility of the element added to modify the surface.

Specifically for Uranium-Nitrogen binary system [2], whose phase diagram is shown in **Figure 1**, the solubility of nitrogen at the melting point of uranium is about 10 ppm [6] [7], while at 2800˚C, the uranium nitride decomposes and forms a liquid phase, containing 4.6 wt% of U. According to the analysis of Bugl J. *et al.* [6], the uranium would form a eutectic with the UN, so that the solubility could be even lower at the annealing temperatures applied for particles surface coating.

Due to the solubility limitations, besides the previous observation that the effectiveness of the UN coating appeared to be small [3], the idea is to attempt the dissolution of nitrogen into UMo alloy in liquid state, which would allow reaching higher solubility of nitrogen and increase the probability to form stable compounds.

Being the centrifugal atomization, the most generalized method and, apparently, the most proper for UMo alloy particles production, the goal of this study is to propose the modification of the surface of UMo particles, during the atomization process, in which the alloy is, by short time, in liquid state. Depending on the feasibility of implementation, this could be a suitable surface modification process for UMo particles, considering industrialization and economic issues.

This paper reports results of reactive atomization conducted under nitrogen atmosphere. This gas is easily available, nevertheless, other reactive gas, such as methane or methane-nitrogen mixture, also could be applied as a way to promote the formation of surface layers, rich in uranium carbide or uranium carbo-nitrides, compounds initially considered as fuel for fast breeder reactor [8].

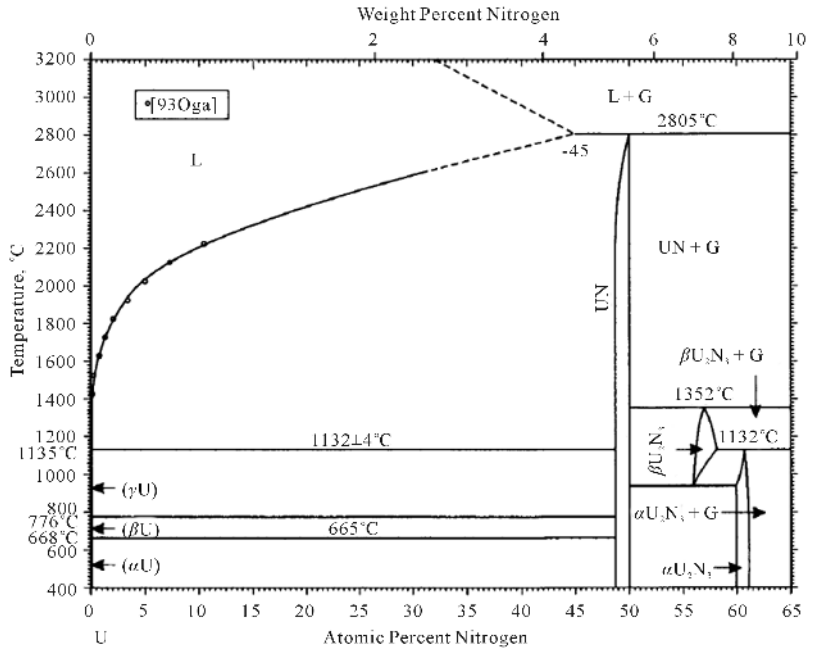

Figure 1. Binary Phase Diagram for uranium-nitrogen [7].

2. Experimental Set-Up

2.1. Atomization Process

The atomization of cylindrical pins to produce UMo alloy particles was carried out in a laboratory scale centrifugal atomizer, described in a previous paper [9]. Since one of the main characteristics of the Rotating Electrode Process-REP is his versatility for the construction of laboratory designs and designs for facilities on a large scale of major importance [10]-[12]. In case of this work, the atomizer chamber is located inside a glove box, capable to operate under controlled atmosphere or rough vacuum. The atomized UMo particles were characterized by SEM-EDS and XRD. The atomization process was carried out with the following parameters (**Table 1**).

2.2. Interaction Test

Out-of-Pile interaction test were conducted using mini compacts prepared with UMo particles dispersed in Al-4wt%Si blend. The total mass of compacts was 0.4 g. To avoid excessive oxidation of samples, the compacts were annealed, at 500˚C by 4 hours, in vacuum and inside a copper envelope. After that, the compacts were mounted in epoxy resin, sectioned and prepared to cross section examination through SEM-EDS microanalyses.

Standard Tyler meshes were used to evaluate the granulometry of UMo particles.

2.3. Miniplates Manufacturing

Using standard methodologies of hot and cold rolling, a set of 6 miniplates was manufactured, with uranium densities of 6, 7 and 8 gU/cm^3. For comparative analysis, a second set of miniplates, with the same densities, was manufactured using UMo particles atomized under argon. QA&QC of miniplates includes; Industrial X-Ray, Ultrasonic Scanning Test, optical densitometry, cladding thickness measurement by Eddy current and cross section examination by SEM and optical microscopy.

3. Results and Discussion

The granulometry of UMo particles atomized under nitrogen and argon atmospheres, selected for miniplates manufacturing, are summarized on **Table 2** and **Table 3**, respectively. The percent of fine particles in the nitrogen atomized alloy was 6.68%, much less than fine particles produced by argon atomization (31.56 wt%). Due to the excess of fine particles, 11.28 grams of material <45 μm were removed, prior to manufacture the miniplates. Considering the particle size range chosen for experimental test (45 and 150 μm), the atomization under argon atmosphere results more efficient for fine particles production. In terms of material recovery, the centrifugal atomization process applied at CCHEN can be considered as high efficiency process, near to 99%, comparable with the results of standard atomization described in [13].

The differences in particle size distribution between the two systems could be related to the presence of dissolved nitrogen in the liquid UMo alloy, which increases the melting point and decreases the density of the alloy. According to centrifugal atomization models, these variations increase the particle diameter [10].

The shape of the UMo particles atomized in nitrogen and argon, inspected by Scanning Electron Microscope and EDS analysis, are shown in the micrographs of **Figure 2** and **Figure 3**, respectively. Some irregular forms are observed, however the spherical morphology is predominant, and this is due to the superficial tension, according to the author K. H. Kim [14], the spherical particles exhibits surface layers, apparently brittle. The high

Table 1. Operational parameters for centrifugal atomization process.

Parameter	Value
Gas Flow (Argon or Nitrogen)	12 liters/minute (welding torch)
Alloy composition	U + 7 wt% Mo (natural uranium)
Pins diameter	8 - 10 mm
Mass of pins	38 - 54 g
Angular speed	40,000 rpm
Welding Machine Current Intensity	60 amps

Table 2. Granulometry of UMo particles atomized in nitrogen.

Lot Identification: U7Mo-REP-NAT-L12			
Tyler Sieve		Material below*	
#	Mesh [µm]	[g]	[%]
100	150	21.84	100.00
170	90	9.81	47.36
230	63	4.66	23.72
325	45	5.18	12.48
Total	-----	41.49	-----

*Excluding the material >150 µm; Total atomized material: 36.11 g + 41.49 g = 77.6 g; Mass of particles >150 µm: 36,11g; corresponding to 46.5 wt% of total; Mass of particles <150 µm: 41,49g corresponding to 53.5 wt% of total; Mass of particles <45 µm: 5.18 g corresponding to 6.68 wt% of total.

Table 3. Granulometry of UMo particles atomized in Argon.

Lot Identification: U7Mo-REP-NAT-L11			
Tyler Sieve		Material below*	
#	Mesh [µm]	[g]	[%]
100	150	22.25	100.00
170	90	6.37	46.13
230	63	4.99	30.70
325	45	7.69	18.62
Total	-----	41.30	-----

*Excluding the material >150 µm; Total atomized material: 7.52 g + 41.30 g = 48.82 g; Mass of particles >150 µm: 7.52g; corresponding to 15.4 wt% of total; Mass of particles <150 µm: 52.58g corresponding to 84.6 wt% of total; Mass of particles <45 µm: 7.69 g + 11.28 g (removed of the Lot) = 18.97 g, corresponding to 38.86 wt% of total.

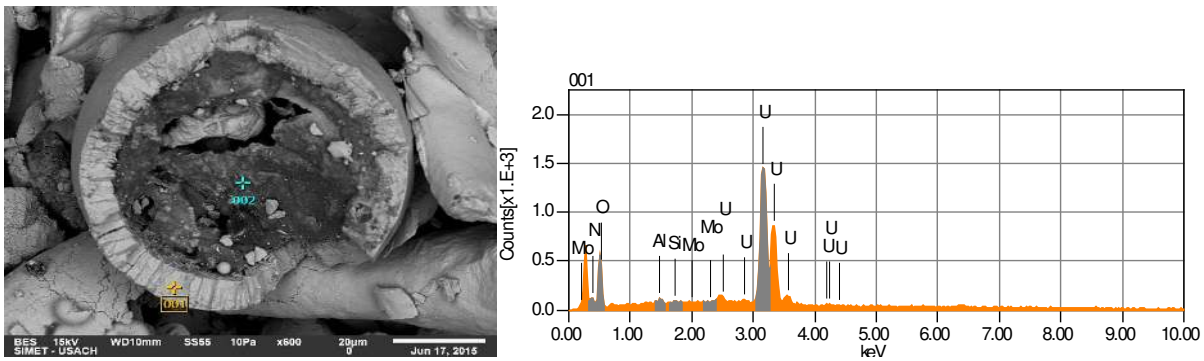

Figure 2. SEM micrographs and EDS analyses of UMo particles atomized in nitrogen.

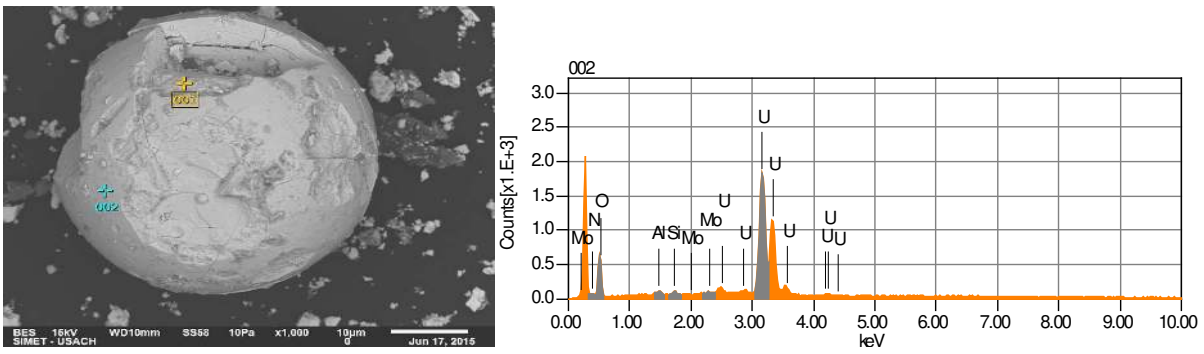

Figure 3. SEM micrographs and EDS analyses of UMo particles atomized in argon.

oxygen content detected by punctual microanalysis on the surface of the UMo particles, verifies the formation of uranium oxide layers, with thicknesses in the order of 5 µm, as is possible to observe in the **Figure 2** (left).

According to the EDS results summarized in **Table 4**, the nitrogen content in the surface is slightly higher for the UMo particle atomized under nitrogen. On the other hand, the nitrogen contents measured by LECO analyzer, were 0.70 and 0.33 wt%, for nitrogen and argon atomization, respectively.

The U-7%Mo particles atomized under nitrogen and argon atmospheres was inspected analytically with X-Ray diffraction in a diffractometer (Shimadzu XRD 6000) with CuKα radiations and divergence and reception slots of 1.0˚ and 0.3˚ mm, respectively. Diffraction patterns of particles atomized in nitrogen and argon atmospheres were done, for theta angles, between 10˚ and 120˚ degrees, with steps of 0.02˚ and 1˚ second. The X-Ray diffraction patterns of atomized U-7%Mo alloy powders are shown in **Figure 4**.

In the UMo atomized particles with sizes below 150 µm, Uγ (bcc) solid solution metastable was detected; this phase can be retained in a metastable state at room temperature by Mo addition and fast solidification [14]. The UO_2 compound was detected in both types of UMo particles; these oxides correspond to superficial oxidation and have a depth of 5 µm approximately, as is possible to observe in the **Figure 2**. The powders atomized in nitrogen atmosphere present, additionally, the compound uranium nitride UN in the surface, which evidence that during the process of atomization, superficial nitriding of the UMo particles occurs. The compound UN was not detected in the sample atomized in argon, according to XRD patterns included in **Figure 4**.

The samples for the interaction test were prepared blending aluminium powder with UMo particles, and then pressing the mix into a die of 8 mm diameter, using a load of 260 MPa. The samples were placed into copper envelopes for interaction annealing test, carried out under vacuum for 4 hours at 500˚C. After that, the samples were mounted in epoxy resin and prepared through metallographic techniques, grinded and polished after each thermal treatment. Optical and Scanning Electron Microscopy—SEM equipped with Energy Dispersed Scanning—EDS microanalyses were used for characterization of fuel particles and interaction layer.

The result of interaction test, included in **Figure 5** and **Figure 6**, reveals the formation of fuel/matrix Interaction Layer (IL). Nevertheless, the content of Al in the center of fuel particle atomized in nitrogen is practically the half than the measured content into the UMo particle atomized in argon (see **Table 5**). No significant differences were observed in the content of others element present at the system.

EDS analyses results evidence the effect of nitrogen as diffusion barrier to avoid Al migration from matrix to inner of UMo particle during U-Al interaction annealing test. The aluminium content into UMo particles atomized under nitrogen is very lower than the content detected into UMo particles atomized in argon atmosphere,

Table 4. Microanalysis results at the surface of UMo particles, corresponding to **Figure 2** and **Figure 3**.

Atomization Atmosphere	Nitrogen (**Figure 2**)		Argon (**Figure 3**)	
Point of analysis	Surface (001)		Surface (002)	
Element	Mass (%)	Atom (%)	Mass (%)	Atom (%)
N	1.4	12.66	0.96	9.04
O	4.6	34.49	4.45	36.76
Mo	0.02	0.02	0.20	0.28
U	93.79	49.97	94.02	52.16

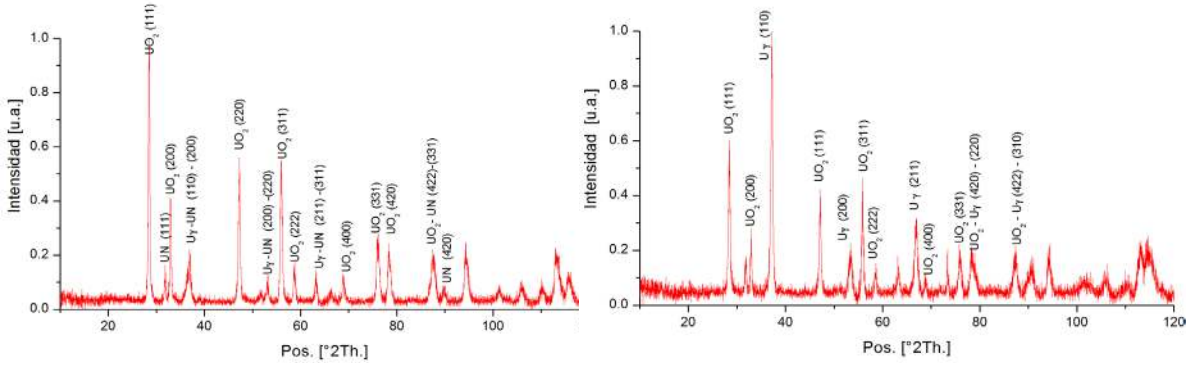

Figure 4. X-Ray diffraction patterns of UMo particles atomized in nitrogen (left) and atomized in argon (right).

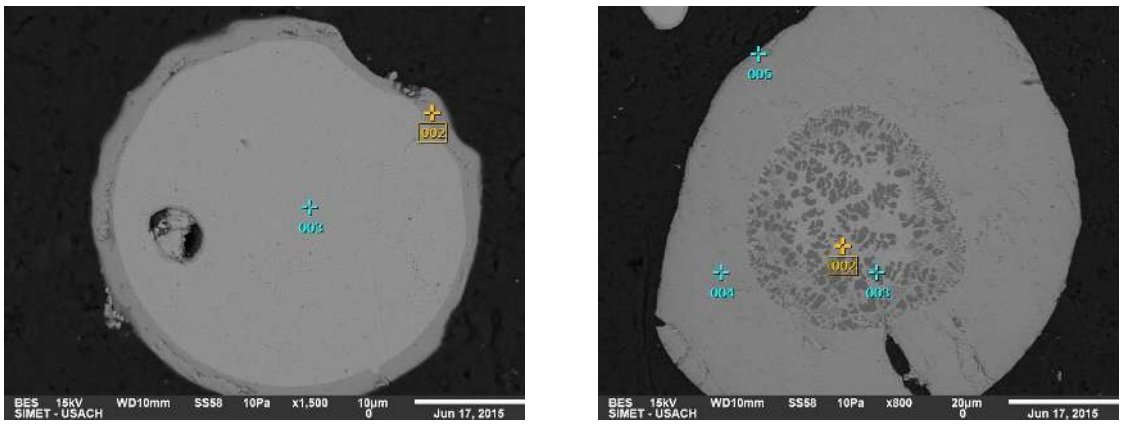

Figure 5. UMo particles atomized under nitrogen atmosphere, after interaction annealing (500˚C/4 hours).

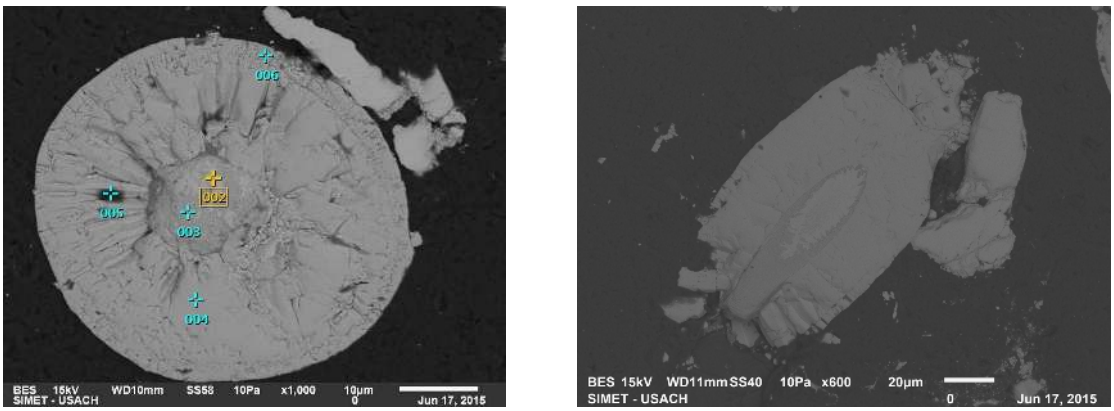

Figure 6. UMo particles atomized under argon atmosphere, after interaction annealing (500˚C/4 hours).

Table 5. Microanalysis results corresponding to the samples showed in **Figure 5** and **Figure 6**.

Atomization Atmosphere	Nitrogen (**Figure 5** right)				Argon (**Figure 6** left)			
Point of analysis	Surface (001)				Surface (002)			
Element (Mass %)	Center (2)	Center (3)	Inter zone (4)	Surface (5)	Center (3)	Center (2)	Inter layer (4)	Surface (6)
N	1.58	1.24	3.17	0.95	1.32	2.23	2.53	0.93
O	1.68	0.93	3.49	3.64	3.90	3.17	5.85	5.27
Al	2.48	1.87	1.77	90.47	3.65	2.20	1.91	89.86
Si	0.06	0.15	0.10	0.41	0.12	0.04	0.06	0.88
U	32.34	82.71	91.37	4.43	90.40	92.17	89.50	2.91
Mo	61.58	13.10	0.11	0.10	0.61	0.19	0.14	0.15

minimizing the probability to promote the formation of U-Al compounds due to interaction, results completely agreed with previous studies [15] [16].

The miniplates were manufactured including blending of UMo + Al-4%Si powders, compacting, assembling (compact, covers and frame), welding, hot rolling, blister test, cold rolling and QA inspections. In **Table 1** is included a summary of fabrication parameters for the complete batch of 6 miniplates.

Table 6 summarizes the SEM-EDS microanalyses results of UMo particles atomized under nitrogen and argon after interaction annealing, whose SEM micrographs were included in **Figure 7**. Regarding to nitrogen contents, it reached up to 7.57 wt% at the center of particle atomized under nitrogen, very higher than 0.84 wt% of nitrogen measured at the center of UMo particle atomized under argon. The results also reveals decreasing of aluminium contents into UMo particles atomized under nitrogen atmosphere; Just 3.77 wt% of Al was the maximum content detected in the center of the particle, very lower than 29.11 wt% of Al measured inside UMo particle atomized under argon.

According to data included in **Table 7**, the total reduction applied to UMo miniplates was in the order of 74%, lower than U_3Si_2 dispersion fuel plates made of, in which the total reduction is approximately 85%. Compared with miniplates made of hydrided powder, the reduction rate and total reduction are slightly higher than those used for atomized powder miniplates [11].

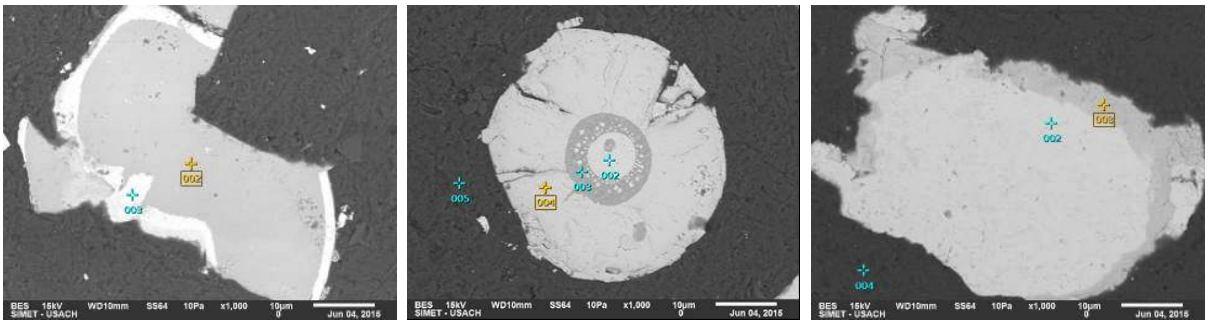

Figure 7. SEM micrographs of Mo particles atomized under nitrogen atmosphere (left and center). UMo Particle atomized in argon (right). All samples after interaction annealing (500˚C/4 hours).

Table 6. Microanalysis results corresponding to the samples of **Figure 7**.

Atomization Atmosphere	Nitrogen (**Figure 7** left)		Nitrogen (**Figure 7** center)			Argon (**Figure 7** right)		
Point of analysis								
Element (Mass %)	Inter layer (3)	Center (2)	Inter layer (4)	Inter zone (3)	Center (2)	Inter layer (3)	Matrix (4)	Center (2)
N	2.80	1.75	7.52	6.14	7.57	0.33	0.55	0.84
O	4.08	1.69	1.91	1.40	0.85	6.81	10.36	1.25
Al	1.64	3.77	1.58	1.49	1.42	29.11	88.81	2.00
Si	n/d	n/d	0.07	0.06	0.10	0.33	0.28	0.09
U	91.29	90.37	88.29	88.99	88.00	63.42	n/d	91.54
Mo	0.18	2.42	0.63	1.92	2.06	n/d	n/d	4.28

n/a: not detected.

Table 7. Manufacturing parameters for UMo dispersion type fuel miniplates.

Miniplate Identification		UMo-97 (argon)	UMo-102 (argon)	UMo-103 (nitrogen)	UMo-104 (nitrogen)	UMo-105 (nitrogen)	UMo-106 (nitrogen)	UMo-107 (nitrogen)	UMo-108 (nitrogen)
Uranium Density gU/cm^3		6.0	8.0	6.0	6.0	7.0	7.0	8.0	8.0
UMo Fuel Mass [g]		5.93	6.40	5.93	5.93	6.18	6.18	6.40	6.40
Mass (Al + 4% Si) [g]		1.52	1.05	1.52	1.52	1.27	1.27	1.05	1.05
Compact Metrology	22.41	22.41	22.41	22.40	22.40	22.40	22.40	22.40	22.40
	18.0	18.0	18.0	18.0	18.0	18.0	18.0	18.0	18.0
	2.47	2.47	2.47	2.80	2.47	2.50	2.64	2.46	2.47
Meat Metrology	86.2	86.2	86.2	86.7	86.5	83.3	83.1	81.9	82.0
	18.8	18.8	18.8	18.0	18.0	18.0	18.0	18.0	18.8
	Thickn. (mm)	0.65	0.65	0.69	0.68	0.67	0.66	0.64	0.64
Meat Volume (cm^3)		1.04	1.05	1.08	1.06	1.00	0.99	0.94	0.99
Volume fraction matrix		0.50	0.39	0.50	0.50	0.45	0.45	0.39	0.39
Miniplate metrology	Length (mm)	130.17	130.21	130.42	130.51	130.40	130.24	130.36	130.21
	Wide (mm)	51.32	50.64	50.92	50.53	50.69	50.63	50.55	50.82
	Thickn. (mm)	1.44	1.44	1.43	1.43	1.43	1.42	1.43	1.42
Starting Thickness (mm)		5.80	5.48	5.80	5.76	5.63	5.58	5.42	5.40
Total Reduction (%)		75.1	73.8	75.3	75.2	74.7	74.5	73.7	73.7

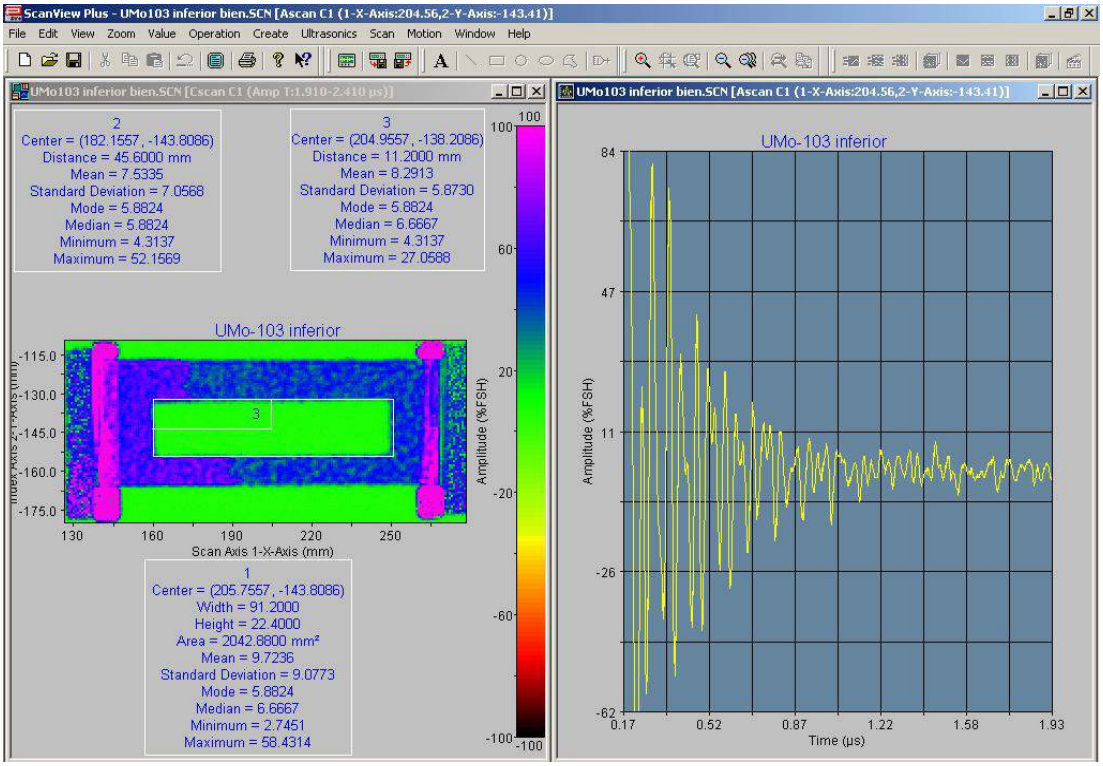

Figure 8. UT inspection of miniplate (UMo-103).

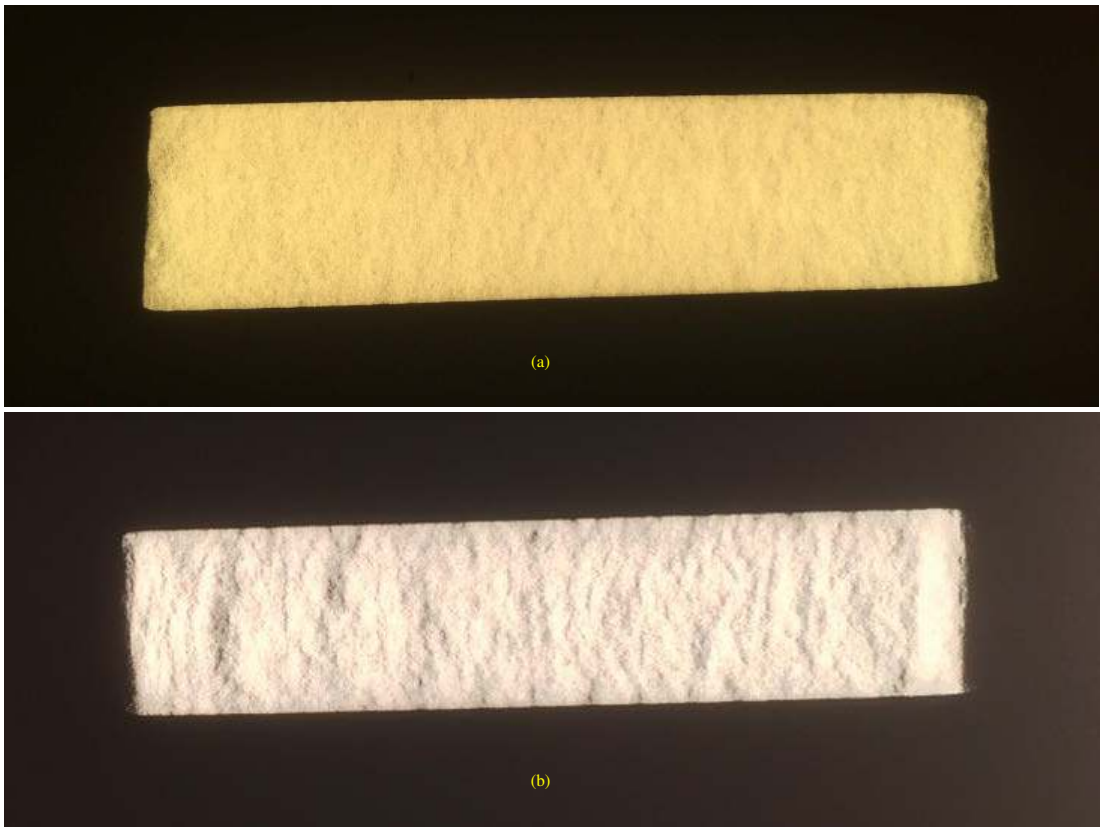

Figure 9. X-Ray images of miniplates (meat zone): (a) UMo-107 (nitrogen), (b) UMo-102 (argon).

The results of ultrasonic scanning test, carried out in eco-pulse mode, reveal the absence of non-bonding areas. Besides, is possible to observe an UT waveform, characteristic of UMo dispersion fuel. Images of UT inspection, corresponding to UMo-103 miniplate is included in **Figure 8**.

Results of industrial X-Ray inspection are shown in **Figure 9**. The radiography (a) corresponds to the miniplate manufactured with particles atomized in nitrogen, where is possible to observe improvement in the homogeneity of particles distribution, compared with the image (b), taken from a miniplate based on particles atomized in argon.

The improvement in the homogeneity can be related with the modification in size distribution of UMo particles. As has been mentioned before, the addition of nitrogen to UMo alloy may produce larger particles and more narrow size distribution. On the other hand, the particles with spherical shape obtained by Rotating Electrode Process are easily segregated, or relocated, during the mixing with aluminium powder and even after, in the matrix, during the rolling process [17].

4. Conclusions

The centrifugal atomization of U-7wt% Mo alloy, conducted under nitrogen atmosphere, was achieved without significant differences in the process, compared with the atomization in argon.

The particle size distribution was the main difference between particles atomized under argon and nitrogen. The presence of nitrogen could increase the melting point and decrease the density of UMo alloy [10]. As a consequence of this, the diameter of particles atomized in nitrogen was increased.

The surface of the UMo particles atomized in argon, analyzed by XRD, reveals the presence of UO_2 and γ-U. In case of particles atomized under nitrogen, the same compounds were detected, and additionally, peaks of uranium nitride UN.

UMo/Al interaction test confirms that the nitrogen addition can decrease but not eliminate this phenomena, nevertheless, the content of Al into the UMo particles was lower in particles atomized under nitrogen atmosphere.

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