Zr OXIDE REMOVAL BY ABRASIVE BLASTING

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ABSTRACT
We have removed the ZrO layer formed on a Zry sample after heat treatment using a simple process known as abrasive blasting or air impelled glass abrasion. Scanning electron microscopy, energy dispersive spectrometry and optical microscopy were the analysis techniques employed to observe the surface after abrasive blasting and the incorporation of glass spheres on the layer that might produce alterations on the chemical properties of the Zry.

Keywords: Zr, blasting, glass, removing.

REMOCION DE OXIDO DE Zr MEDIANTE GRANALLADO

RESUMEN
Mediante el uso del proceso de granallado con partículas de vidrio se ha removido la capa de óxido de Zr producida durante un tratamiento térmico formado sobre una muestra de Zry. Las técnicas analíticas empleadas para la observación de la superficie y de las partículas de vidrio adheridas sobre la misma luego del granallado fueron: SEM, EDS y microscopía óptica.

Palabras clave: Zr, granallado, vidrio, remoción.

INTRODUCTION

The zircalloy (Zry) is a trade mark of Zr alloys. Zr has very low absorption cross-section of thermal neutrons (0.18 barn), high hardness, ductility and corrosion resistance, for this reasons is mainly used in nuclear technology, for example as cladding of fuel rods in nuclear water power reactors. The typical composition of nuclear-grade zirconium alloys is more than 95 weight percent zirconium and less than 2 percent of tin, niobium, iron, chromium, nickel, and other metals; added to improve mechanical properties and corrosion resistance. The study of new fuels for research reactors intends to reduce the employ of ^{235}U and with this objective we have started to manufacture UMo

Fig. 1. Blasting camera

compact fuels consisting of a thin plate of an $U_{93}Mo_7$ covered with two Zry slices. After welded and heat rolled in open air, to diminish its thickness, we observed a dark Zr oxidized layer. This oxides have low thermal conductivity and it is a big problem for heat transfer during nuclear reactor operation and must be removed. The Zr oxides hardness is very high and rather difficult to eliminate, they resists to almost all acids and they are so firmly adhered that oppose ordinary mechanical abrasion. In order to study the removal of the oxide surface, we have prepared a rectangular Zry dummy sample which was annealed in open air simulating to be a nuclear fuel cell. The oxidized layer was eliminated in few minutes using a simple and well known device called sand erosioner or abrasive blasting, [1,2]. It consists of a camera, similar to a glove box, Fig. 1, where pieces to be surface treated are introduced and suffer the action of a beam of hard spherical glass micro particles impelled by pressurized air.

MATERIALS AND METHODS

We employed a Pecinelec7 equipment using an air flux of 1 m^3/min at 70 Ncm-^{2} for impelling the glass micro spheres, Fig. 2. Glass particles 90 to 150 μm in size were introduced by Bernoulli effect into the air flux reaching an estimated velocity of 8000 mms^{-1} and a kinetic energy of 2.9 10-^{17} J. The studied material consisted of one Zry slice 4 cm x 3 cm x 0.5 cm in size. After rolled and annealed in open air, a black oxide layer about 15 μm thick was formed, Fig.3. The piece was introduced into the box grasped by glove covered hands. The air impelled glass spheres were hand directed to the sample in order to scan the entire sample surface. The surface suffered the action of the spherical impinging particles, Fig. 2. Sparks were observed during the process due to swift oxidation of Zr after the oxides had eroded.

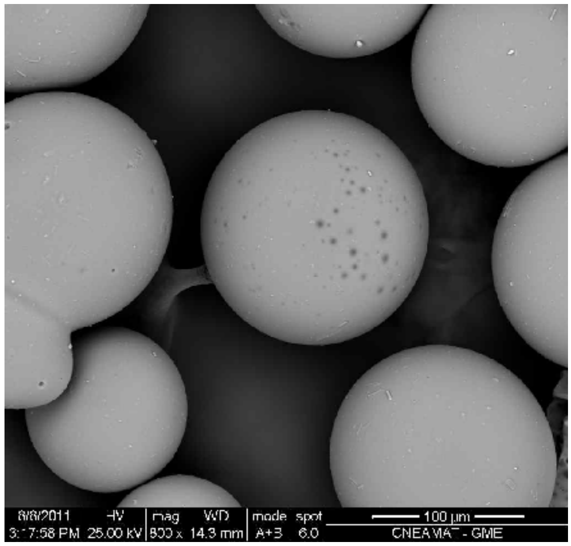

Fig. 2. Glass particles at high magnification.

For hardness measurements the sample was included in acrylic to show the perpendicular surface to the affected one.

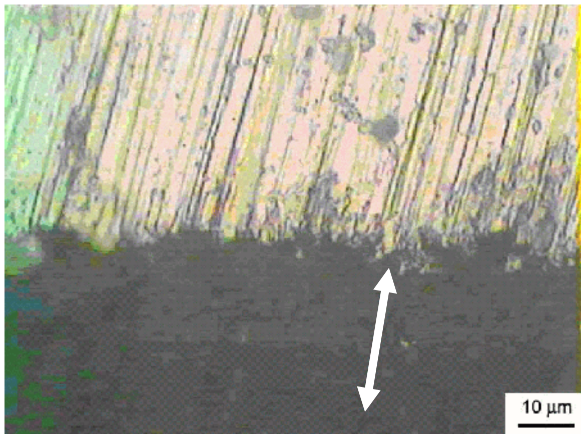

Fig. 3. Cross section micrograph of the oxidized surface, arrow indicates thickness of Zr oxide.

RESULTS

The black oxidized layer shown in Fig. 4 was removed in few minutes by abrasive blasting and a grey metallic one appeared as can be seen in Fig. 5, where a track was produced with a steel file to prove that the hard oxide has been removed.

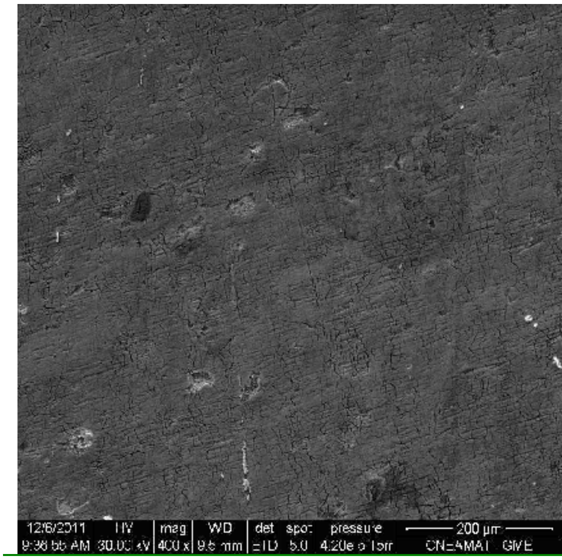

Fig. 4. SEM of the oxidized layer.

Table 1. Measured Hardness.

	Hardness Hv 0.015)
On the matrix	278-300
On the oxide layer	440

The surface aspect was the usual of glass erosioned metals, Figs 6 and 7. In the SEM backscattered image in Fig. 5, glass particles adhered to the surface can be seen as dark spots while the bright zone, (the previously mentioned light track) corresponds to Zry. The particles' average composition obtained by EDS was 60 wt % and Ca 40 wt % (elements of atomic number under 11 were not detected) Fig.8.

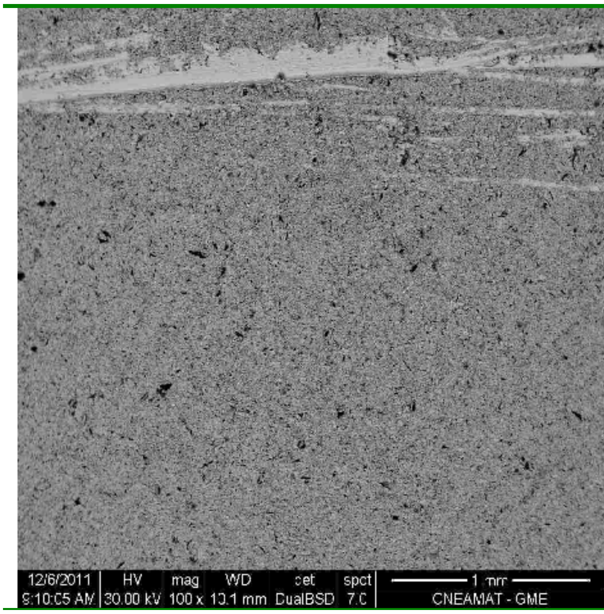

Fig. 5. SEM with backscattered electrons showing the file trace and glass particles, see the dark points.

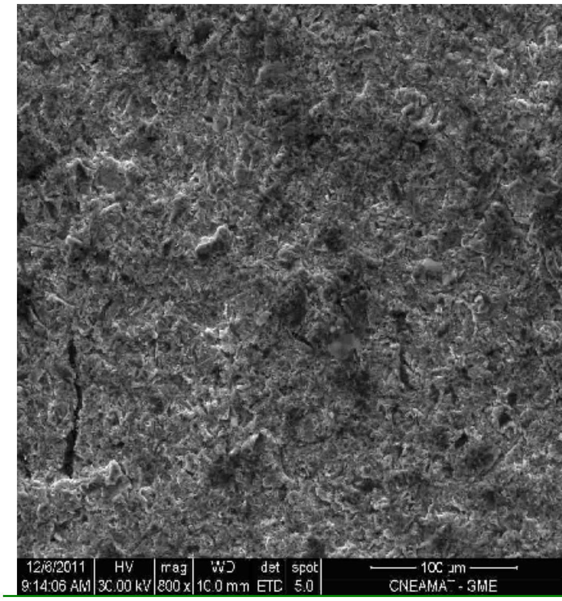

Fig. 6 Aspect of the glass erosioned layer

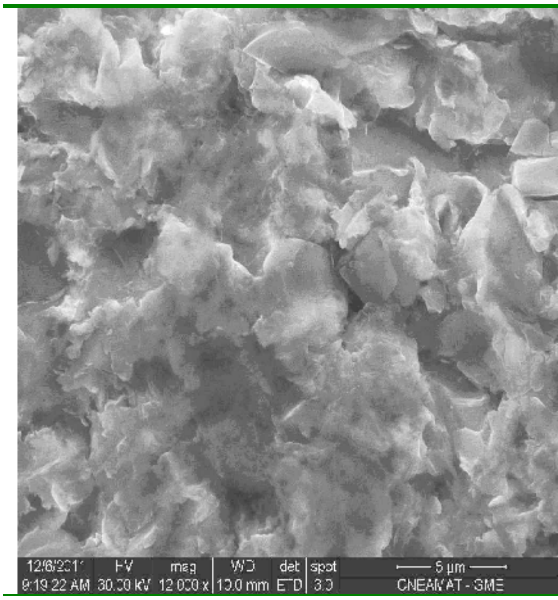

Fig. 7 Aspect of the glass erosioned layer at higher magnification

Micro-hardness, using Hv 0.015 g load and following ASTM E 384 norm were taken on both the oxidized layer and the matrix, see Table I. The hardness measurements were made on the cross section sample.

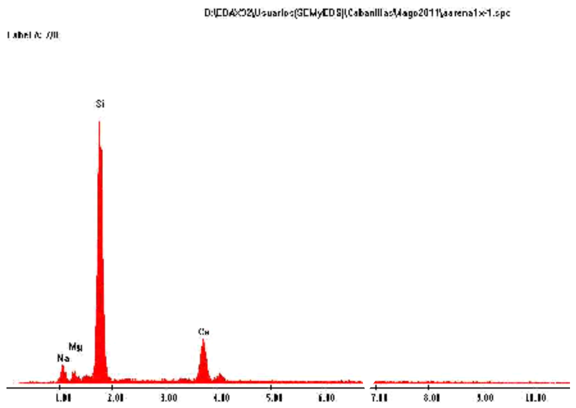

Fig. 8. EDS of glass particles.

DISCUSSION

Each air impelled glass particle, with high kinetic energy impinging on the surface, produces a high tension, which, being higher to the fracture tension of Zr oxides, would crush them and would remove the chips.. Once the first chip have been detached the next are easier to eliminate, so the hard oxide layer is weakened and completely removed. Without the oxide layer, the glass spherical particles produce an indentation of a few μm^2 like those made by the spherical extreme of a rivet hammer, obtaining the surface shown in Fig. 7. Thus the kinetic energy and the hardness of glass particles are the cause of oxide removal, [3,4].

The process is similar to that of a water cutting machine, [5] where the cutting is produced by irregular abrasive particles, in this case impelled by water a water flux at very high pressure of about 3000 bar. But the surface panorama is quite different in these cases, scratches appear when water cutting occurs while in the case of blasting the surface looks rather flaky, Fig. 7, meaning that a pein hammering like process has occurred with spherical bullets impinging on the metal surface instead of a ball pein hammer. The pein in this case is rounded and metal workers shape metals and close rivets with this type or tool.

CONCLUSIONS

1) The abrasive blasting process is appropriate to remove the Zr oxides and other deposited materials, the equipment could be got commercially and do not presents chemical risks, the operation is done in a closed chamber.

2) After the employment of this process the surface must be thoroughly cleaned because loose particles may remain on the surface.

3) Care should be taken with material which can suffer plastic phase transformations.

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REFERENCES

[1] Mead W. H. (1957) U.S. patent 2,810,991.

[2] Hey A., Harriague S. and Fabbri S. (2006) "Descontaminación mecánica de componentes nucleares" *Asociación Argentina de Materiales Registro ISSN 1668-4788* 3 www.materiales-sam.org.ar (1):3-14.

[3] Achtsnick M., Geelhoed P.F., Hoogstrate A.M.and Karpuschwsk B.(2005) "Modelling and evaluation of the micro abrasive blasting process" *Wear*, 259: (1-6):84-94.

[4] Bhushan, B. and Gupta, B.K. (1991) *Handbook*

of Tribology: Materials, coatings, and surface treatments, McGraw-Hill, N.Y. Chapter 6.

[5] Cabanillas, E.D. (2010) “Micro and nano particles produced by water jet abrasion” *Acta Microscopica*, 19 (1):105-108.