The Metallography of Mixed Oxide Fuel (M.O.X. Fuel) and Canning Materials for Thermal Reactors

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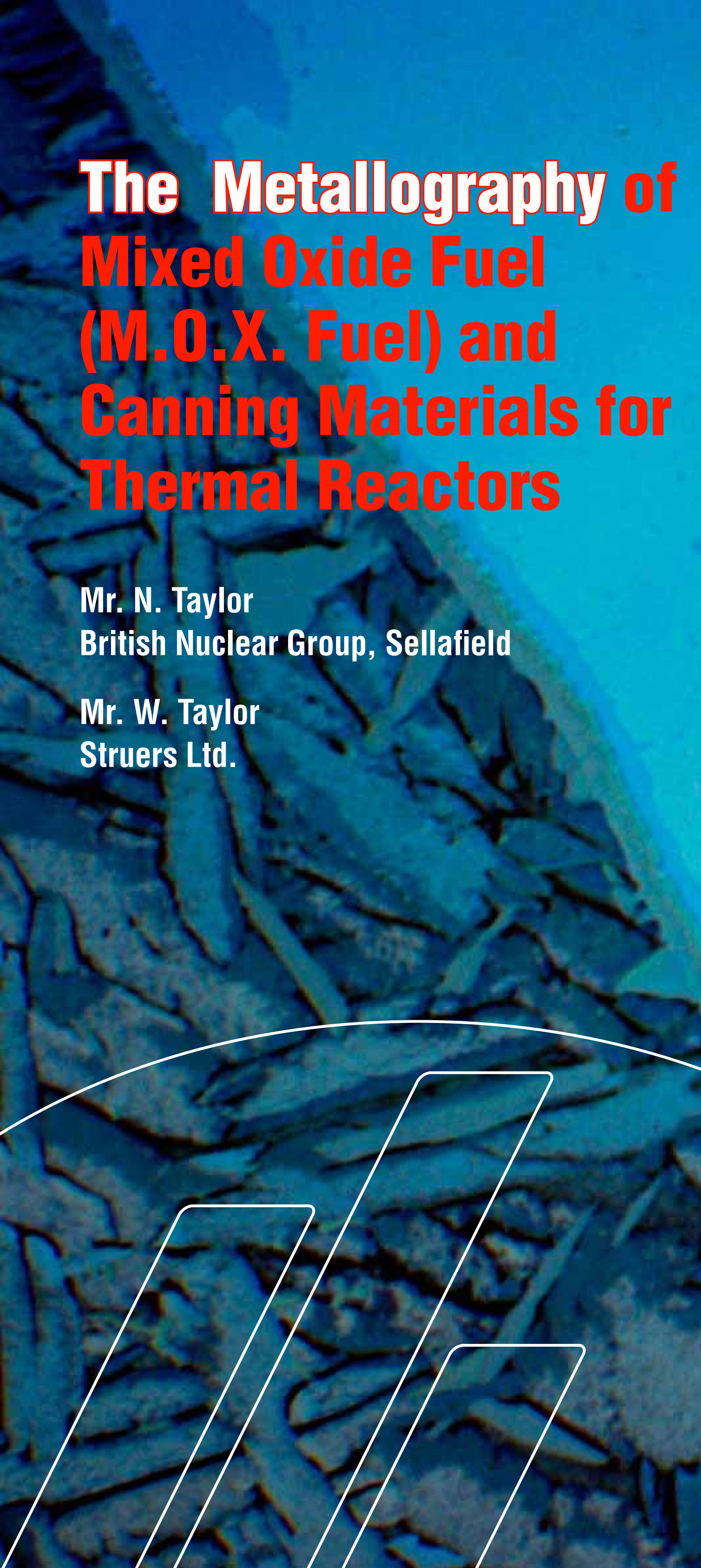

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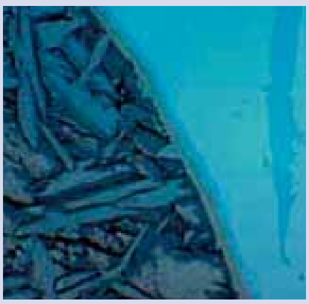

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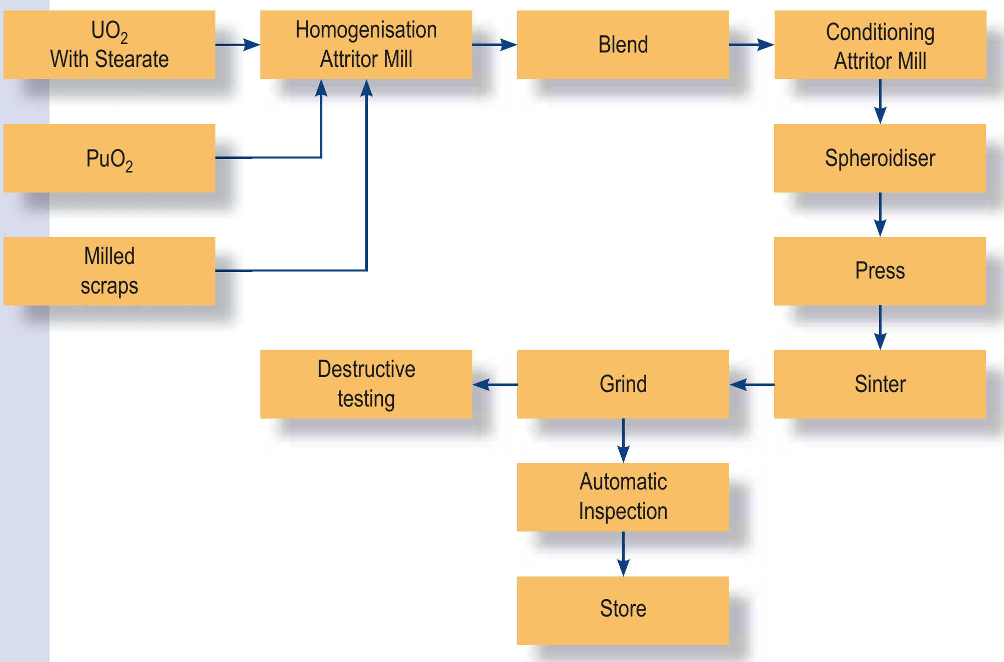
Introduction

The Sellafield Site on the North West coast of England is a global centre for the re-processing and manufacture of fuel for various types of Nuclear Reactors.

The site, on which approximately 10,000 people are employed, is owned by the Nuclear Decommissioning Authority, and is operated by British Nuclear Group. Within the Sellafield complex, the Sellafield Mixed Oxide Plant (SMP) is a large facility attached to the Thermal Oxide Reprocessing Plant (THORP). The Sellafield MOX Plant has been in operation since 2002. Its purpose is to manufacture Mixed Oxide Fuel, and in the manufacture of this fuel, the process utilises small amounts of Plutonium from reprocessed Uranium fuel assemblies.

M.O.X. Fuel

Mixed Oxide Fuel is a mixture of Uranium and Plutonium Dioxide powders in specific ratios. The Plutonium is recovered during the reprocessing of spent Uranium fuel assemblies, and then, this recovered Plutonium is mixed with natural or depleted Uranium Dioxide powder in various process vessels, to produce a homogeneous mixture of Uranium and Plutonium Dioxides.
This powdered feedstock is then pressed into small cylindrical pellets.



The milled, powdered feedstock is tumbled to form granular agglomerates termed spheroids. These spheroids form a close packed array when fed into the pelleting die. At this stage the grains are separated by pores which occupy 30-40% of the pellet volume.
During the subsequent sintering operation at temperatures in excess of 1700°C, the porosity is reduced, density increases, and grain size and grain shape changes occur. These changes are required to transform the friable green pellet into a dense solid ceramic.
After the sintering operation, representative samples of full pellets are submitted to the S.M.P. Analytical Laboratory.

S.M.P. Analytical Laboratory.

The work of the laboratory is divided into three main areas of interest.

Fuel pellets

Dimensional analysis
Ceramography

Fuel rod end caps

Metallography (weld assessment)

Fuel Pellets

Dimensional analysis and ceramography are carried out within the shielded environment, and each individual activity in the process takes place within its own specific glove box.

Pellet Ceramography (microscope)

It should be noted that during 1997/1998 at the design stage of this project, the standard Struers Accutom-5 cutting machine, the RotoPol-22/RotoForce-4 grinding/polishing machines and the Multidoser dosing system were each modified by the consulting engineers responsible for the project. The net result of these modifications was that all control functions were removed to the outside of the glove box.

Ceramography
The glove box activities, as outlined below, are laid out to give an efficient work flow through the Laboratory.

1. Dimensional Analysis	**2.** Encapsulation Glovebox	**3.** Cutting Glovebox Accutom-5	**4.** Grinding Glovebox RotoPol-22 RotoForce-4	**5.** Fine grinding Glovebox RotoPol-22 RotoForce-4	**6.** Final polish and chemical etch glovebox
		10. Pellet Recovery	**9.** Storage	**8.** Autoradiography	**7.** Microscopy

Encapsulation
The first stage in the process is the encapsulation stage. A fuel pellet is placed in the mould with its longitudinal axis parallel to the mould base. Conventional 40 mm cold mounting moulds are used and Struers SeriFix is the resin of choice for the following reasons:

a) The clarity of the resin facilitates alignment of the mount for the subsequent cutting operation
b) It has a relatively short curing time (approx 30 min.)
c) It presents no difficulties on cutting
d) The fuel pellets can be easily recovered from the resin using liquid Nitrogen

Cutting
After curing, the encapsulated specimen is moved to the cutting glove box for sectioning along its the longitudinal central axis. Cutting is carried out on a Struers Accutom-5 using a 430CA cut-off wheel. Apart from the control panel being removed to the outside of the glove box, the only modification to the standard machine is that the wheel and its flanges are retained by a wing nut. This makes the task of changing the cut-off wheel easier.

In any one glove box the volume of water is strictly controlled, and because of this, the cut-off wheel is rotated at 300 rpm. Wheel rim cooling is effected by means of a shallow trough of water situated below the wheel.
Even with these restrictions the encapsulated sections are cut in 10 to 15 minutes.

Mechanical preparation
Grinding and fine grinding stages are carried out on two RotoPol-22 / RotoForce-4 machines using single sample mode. Again all controls for the RotoPol and the RotoForce have been removed to the outside of the glove box.

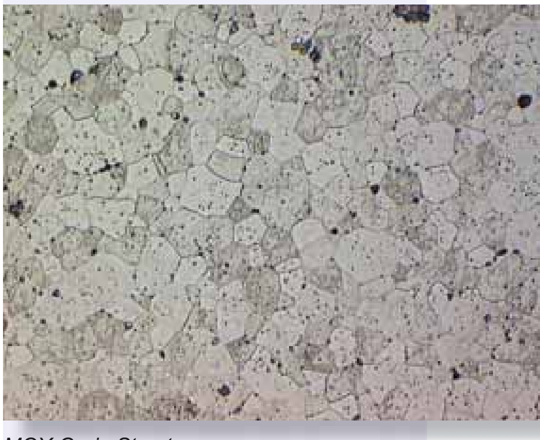
An outline of the method for fuel pellets is provided below. This preparation method was developed in conjunction with Struers and was developed to satisfy the following criteria:

a) Polished sections of a consistent quality
b) Minimum of manual input. e.g. SiC paper changes
c) Minimum of waste products
d) Ease of use i.e. magnetic disc system

Sample preparation gloveboxes

Surface	Glove box	Grit/grain	Rpm	Force	Time min.	Direction	Lub.
MD-Piano	4	220	150	30	2.00	Contra	Water
MD-Allegro	5	6 µm Poly.	150	30	5.00	Comp	Green
MD-Pan	5	6 µm Poly.	150	35	5.00	Comp	Green
MD-Chem	6	OP-S	150	30	1.50	Contra	

Note – Water and lubricant are each dosed to the grinding and fine grinding cells in carefully controlled volumes by means of a Struers Multidoser, which is placed outside of the glove box.
Polishing is effected on the MD-Chem cloth using OP-S activated with an addition of 5% by volume of Hydrogen Peroxide (30vols).
Manual dosing of OP-S is used for the polishing cycle.


MOX Grain Structure

Microscopy
The operational performance of the manufactured fuel is influenced by numerous properties including:

- Fuel density
- Microstructure
- Resintering activity

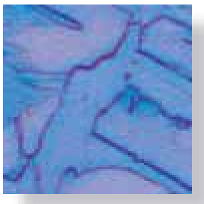
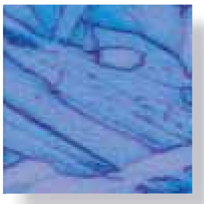
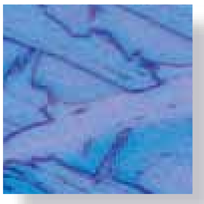
In the as polished condition the size, content and distribution of porosity are measured both comparatively and as an area fraction using image analysis.
The presence, size and distribution of any metallic inclusions are also established at this stage.

Each polished pellet is then exposed to a contact auto radiograph on a colour film; after controlled exposure to the alpha radiating fuel pellet, the film is developed and assessed for Plutonium homogeneity, and Plutonium particle size and enrichment.
It is important in this respect that the polished section has minimum relief in order that the size of the discreet Plutonium particles is not exaggerated.
After chemical etching, the average grain size is established by the linear intercept method in line with the requirements of ASTM E112. This data is recorded.

Fuel rod end caps

Another function of the laboratory is to carry out quality control checks on the T.I.G. (Tungsten inert gas) weld process for fuel rod end cap/cladding welds. These are manufactured from Zirconium based alloys; because of their high corrosion resistance combined with a low neutron cross-section.
No shielding is required for this work.

Cutting
Each end cap is sectioned along the longitudinal central axis on an Accutom-5 using a 457CA cut-off wheel, rotational speed 3.000 rpm.

Mounting
The sections are then cold mounted in Struers CitoFix in 40 mm diameter moulds.

Mechanical Preparation
The sections are prepared on a Struers RotoPol-22 / RotoForce-4 using the method outlined below:

Surface	Grit/grain	Rpm	Force	Time min.	Direction	Lub.
MD-Piano	220	150	25	2.50	Contra	Water
MD-Primo	220	150	20	2.00	Comp	Water
MD-Pan	DiaPro Plan	150	35	4.00	Comp	
MD-Chem	OP-S*	150	30	4.00	Contra	

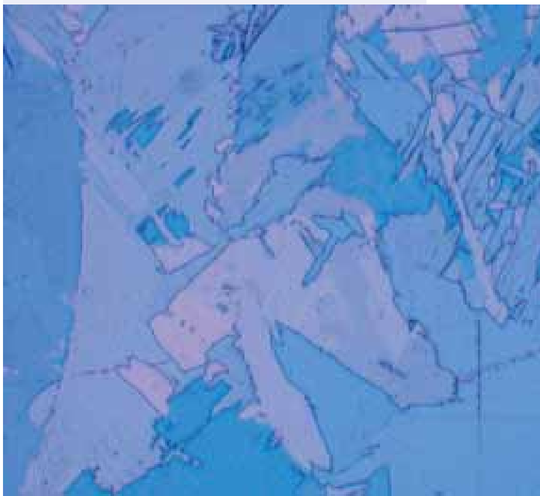
Note – OP-S with an addition of 5% Hydrogen Peroxide (30vols)

Microscopy
In the as polished condition the following checks are made

a) Measurement of cladding wall thickness
b) Measurement of weld concavity
c) Size of porosity in the girth weld
d) Size of porosity in the closure weld
e) Presence of any inclusions

After etching in Kroll's reagent the following measurements are made

a) Depth of fusion on closure weld
b) Depth of penetration on girth weld
c) Depth of undercut on girth weld
d) Depth of fusion on girth weld
e) Axial penetration on girth weld


Weld 50 x pol

Conclusions

The mechanical preparation of materials within the shielded environment presents a number of challenges viz:

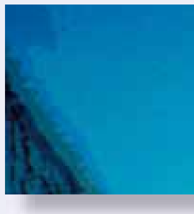
a) **To provide polished sections of a reproducible high quality.**
The degree of automation used for materials preparation in this laboratory was a first for the UK nuclear industry and from the development stages up to the present time there has been close co-operation between British Nuclear Group and Struers. It is now possible to have complete control of all the preparation parameters such that polished sections of a high quality are consistently produced.

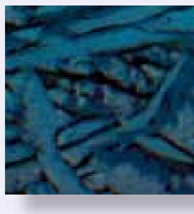
Each of the operators has been provided with comprehensive training on the operation of the equipment as well as attending a Professional Metallographer Course. This training is updated regularly.

b) **To minimise the manual input to the processes within the confines of the glove box**
Semi-automatic preparation equipment and the use of long life consumables e.g. MD Piano, in conjunction with the modifcations made to standard equipment during the pre commissioning stage, has ensured that manual input to the processes is minimised.

c) **To provide a preparation system that is mechanically reliable within the glove box environment**
Mr. N. Taylor, Laboratory Manager, ''Approximately 2,500 fuel pellet sections have been prepared since the Laboratory was commissioned, and there has been no significant mechanical or electronic failures associated with the preparation equipment. The reliability of the system and the consistency of polish have exceeded our expectations.''

d) **To provide an economic preparation method in terms of cost/section and time**
The ability to control the preparation parameters viz force, time, dose rate etc., allows short preparation times and most economic use of abrasives. The use of durable grinding/polishing surfaces optimises consumable life.
MD-Piano approx. 120 sections.
MD-Allegro approx. 200 sections.





e) **To minimise the amount of contaminated waste generated in the preparation process**
In preparation methods which make use of Silicon Carbide papers, much waste is generated in the form of spent grinding paper, and in an industry constantly looking to reduce waste generation, it was important that the preparation method should minimise the amount of waste produced.
The developed method makes no use of Silicon Carbide paper; grinding is carried out on long life, durable grinding surfaces.