# CHARACTERIZATION OF $Y_2O_3$ AND $La_2O_3$ NANOPARTICLES IN W ODS ALLOYS BY SMALL ANGLE NEUTRON SCATTERING

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## INTRODUCTION

W and its alloys are very promising materials for making plasma facing components (PFC) in the future fusion power reactors [1,2]. The properties that make W a suitable material for building PFCs are its high melting point, good thermal conductivity, high thermal stress resistance, low tritium retention and high temperature strength. However, its relatively high ductile-brittle transition temperature (DBTT), in the range 473-673 K, and its recrystallization temperature (RCT), around 1500 K [3], limit the operating temperature range of those W components with structural functions. Nevertheless, the DBTT and RCT for W can be improved by addition of some impurities. The current He-cooled divertor designs are considering a thermal armor of sintered W tiles joined to thimbles of oxide dispersion strengthened (ODS) W alloy. These ODS alloys have to be properly joined to sintered W tiles; besides having a low DBTT and a high RCT.

Recently, W-1$Y_2O_3$ and W-1$La_2O_3$ alloys have been produced by mechanical alloying and subsequent consolidation by hot isostatic pressing [4,5]. The dispersion of nano-particles of $Y_2O_3$ or $La_2O_3$ in the matrix enhances the strength and fracture toughness of W at high temperature. This improvement of mechanical properties of the ODS W alloys appears to be related to the characteristics of the oxide dispersion. Therefore, it would be very useful to study the distribution of the oxide nano-particles in the ODS tungsten alloys. Small angle neutron scattering (SANS) is a suitable technique to analyze the morphology and size distribution of the oxide nanoparticles in ODS-W. The main advantage in using SANS is that quantitative results of the particle dispersion are obtained by averaging over a large sample volume. In this work, the morphology and distribution of particles of $Y_2O_3$ and $La_2O_3$ in W-1$Y_2O_3$ and W-1$La_2O_3$ alloys have been investigated.

## FABRICATION OF THE ALLOYS

**Target compositions:**

- W-1% wt $Y_2O_3$
- W-1% wt $La_2O_3$

**Powder blends:** W: 99 % pure, ∅<5 μm. $Y_2O_3$ , $La_2O_3$ : 99.5 % pure, ∅ 10-50 nm.

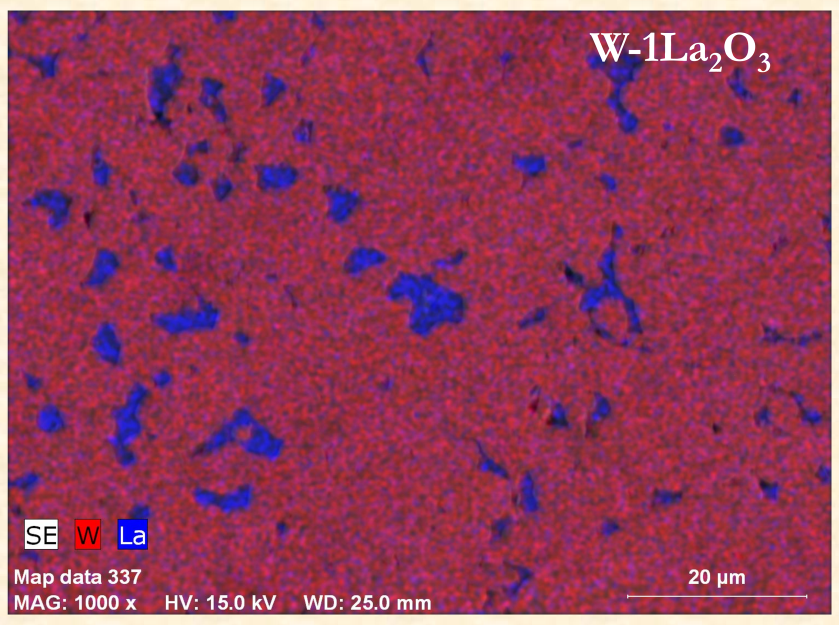
→ **Blending:** 4h in a Turbular mixer

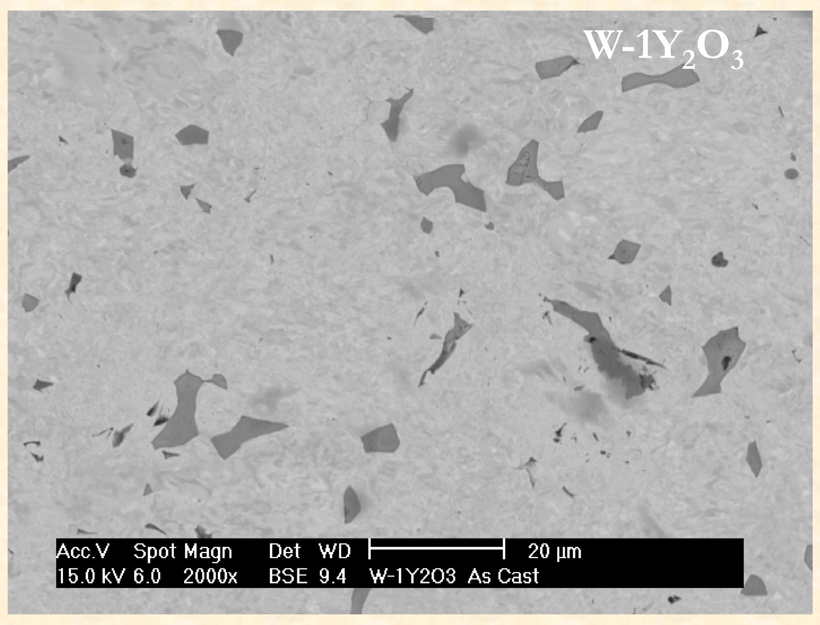
→ **Milling:**
- Ar atmosphere.
- WC vessel and WC balls. Balls to powder ratio 4:3.
- 20 h at 400 rpm.

→ **Canning:**
- Powders packed in 304 stainless steel cans.
- Degassed at 673 K for 24 h.

→ **HIP:** HIP at 1573 and 200 MPa for 2h.

## SCANNING ELECTRON MICROSCOPY



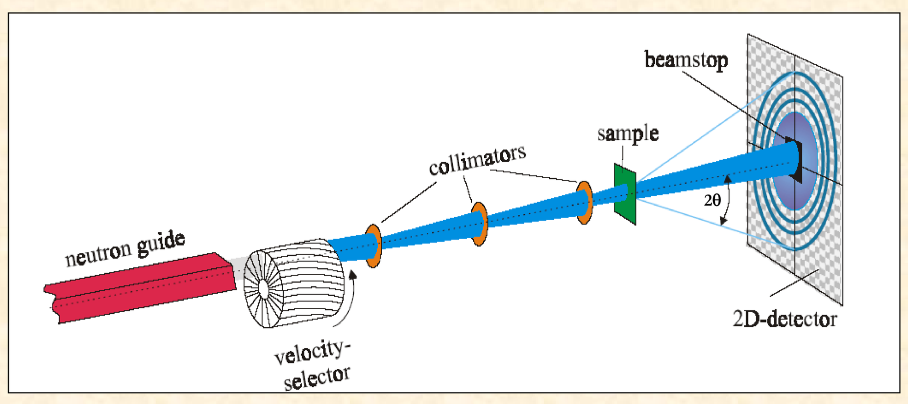

**Mean results:**

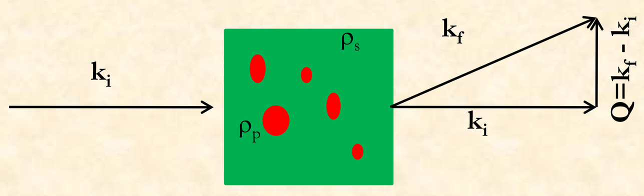
**1.-** After the milling process the $Y_2O_3$ and $La_2O_3$ particles seem to be alloyed with the tugnsten matrix.

**2.-** The consolidation process by HIP produces segregation of Y and La rich oxide pools with dimensions of less than 10 μm at some interstices between W particles.

**May oxide nanoparticles exist dispersed in the W matrix?**

## SMALL ANGLE NEUTRON SCATTERING




$$Q = \frac{4\pi}{\lambda}\sin\theta$$

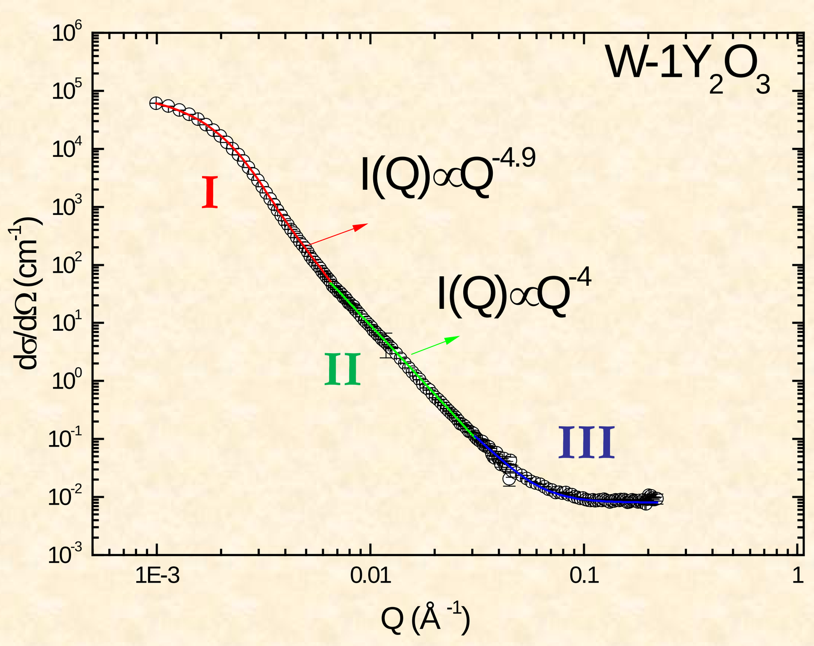
In a SANS experiment, the differential cross-section, $I(Q)$, in terms of the momentum transfer, $Q$, is determined by:

$$I(Q) \propto (\rho_p - \rho_s)^2 P(Q) S(Q)$$

Particle form factor — Structure factor. Its value is 1 for a dilute system

ρ is the scattering length density

The SANS measurements were performed at the instrument KWS-1 in the FRJ-II reactor (Jülich-Germany). The experimental wave length was 7 Å, and three samples to detector-distances of 2, 8 and 20 m were employed. This let us cover a $Q$-range of 0.002-0.14 Å$^{-1}$, corresponding to direct space distances over the range 50-3100 Å.




Three different regimes at different length scales are observed in the scattering curve $I(Q)$. If it is assumed three independent size levels in the microstructure, it can be fitted by three independent Beaucage functions:

$$I(Q) = \sum_{i=1}^{3}\left( A_i e^{-\frac{Q^2R_i^2}{3}} + B_i\left\{\frac{\left[erf\left(QR_i/6^{1/2}\right)\right]^3}{Q}\right\}^P \right)$$

From the Porod's region, the specific surface is given by:

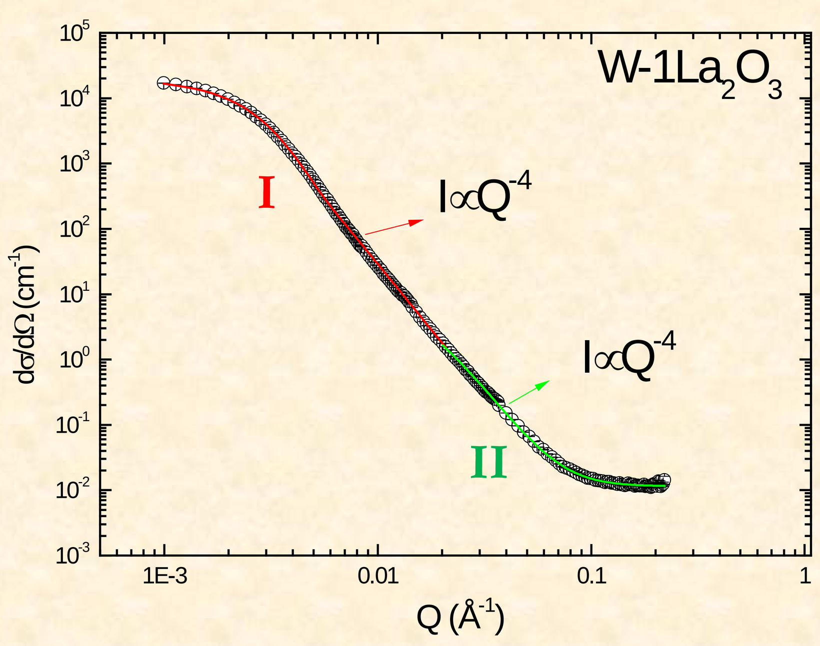
$$\frac{S}{V} = \frac{lim_{q\to\infty}\left(Q^4 I(Q)\right)}{2\pi(\rho_p-\rho_s)^2} = \frac{B}{2\pi(\rho_p-\rho_s)^2}$$

| Level | $R_i$ (nm) | $d_i$ (nm)* | $S/V$ (cm$^{-1}$) | (% wt.)* |
|---|---|---|---|---|
| I | 116(6) | 300(15) | | 0.49 |
| II | 44(1) | 114(1) | 3806 | 0.048 |
| III | 13.0(1) | 34(1) | 4177 | 0.011 |

$R_i$: Radius of gyration.

* Assuming a spherical shape for the particles.

- **For level I, the Porod exponent, $P$, is 4.9. It indicates a diffusive surface. Some W-Y-O compounds are formed in the boundary between the particles and the W matrix.**
- **For Levels II and III the Porod exponent is 4. It implies a sharp smooth surface between the particles and the W matrix.**
- **For level III the average size is around 34 nm. The initial size for the $Y_2O_3$ particles was in the range 10-30 nm.**




Two different regimes at different length scales are found along the scattering curve $I(Q)$. It can be fitted by two independent Beaucage functions:

$$I(Q) = \sum_{i=1}^{2}\left( A_i e^{-\frac{Q^2R_i^2}{3}} + B_i\left\{\frac{\left[erf\left(QR_i/6^{1/2}\right)\right]^3}{Q}\right\}^P \right)$$

| Level | $R_i$ (nm) | $d_i$ (nm)* | $S/V$ (cm$^{-1}$) | (% wt.)* |
|---|---|---|---|---|
| I | 75(2) | 190(5) | 46250 | 1.8 |
| II | 17.0(1) | 44(1) | 55997 | 0.18 |

$R_i$: Radius of gyration.

* Assuming a spherical shape for the particles.

- **For levels I and II, the Porod exponent, $P$, is 4. It implies a smooth surface in all cases.**
- **For level II the average size is around 44 nm. The initial size for the $La_2O_3$ particles was in the interval 10-30 nm.**
- **Compared to W-1$Y_2O_3$, W-1$La_2O_3$ exhibits a finer particle size distribution.**

## CONCLUSIONS

1. The SANS measurements in the size range explored (5-300 nm) have revealed the presence of a trimodal size distribution of oxide particles in the W-1$Y_2O_3$ alloy, and a bimodal one in W-1$La_2O_3$.
2. The finest populations of particles have mean sizes of 34 and 44 nm for W-1 $Y_2O_3$ and W-1 $La_2O_3$, respectively, and an estimated fraction of rather less than 1% mass.
3. The value of the Porod exponent for the different levels found along the scattering curves is 4 except for level I for W-1$Y_2O_3$. In this case, it is 4.9 indicating a diffusive surface for the corresponding population of particles.

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