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TEM Study of mechanically alloyed ODS powder

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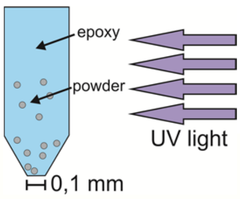
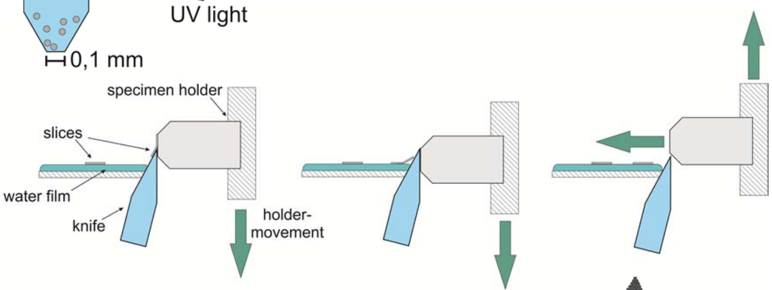
Introduction

The production route for these ODS materials is expensive and it usually takes a long time till a new alloy can be fully characterized. By examining the alloyed powder after milling, a major step can be validated before running through the complete production process. The analyses of the powders need to be done by transmission electron microscopy (TEM). However, preparation of powder samples for TEM is usually very time and cost consuming.
To be fully transmitted by the electron beam, the thickness of particles needs to be thinner than 100 nm, which is a magnitude smaller than the average diameter of powders usually used for powder metallurgy (typically around 10 to 100 µm). In this present work, an alternative existing method for biological and tissue samples was adapted and is described in the following.

Methods and Materials



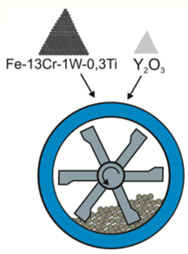
The powder is mixed with UV-hardening epoxy and quilled to have a homogeneous distribution. The holder moves up and down and a diamond blade cuts off very thin slices (around 20 nm thickness).



Experimental

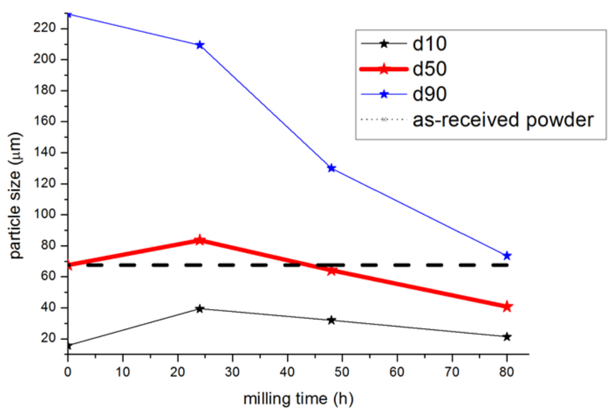
To validate this TEM preparation technique, a small experimental series with the same powders and different milling times was done.

Argon-gas-atomized steel powder was mixed with Y_2O_3 and milled for different times.



Elements	Cr	W	Ti	Y_2O_3	Fe
Base alloy	13	1	0.3	-	bal.
ODS alloy	13	1	0.3	0.3	bal.
milling-times: 80, 48, 24 hours					

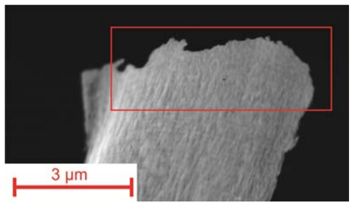
Results



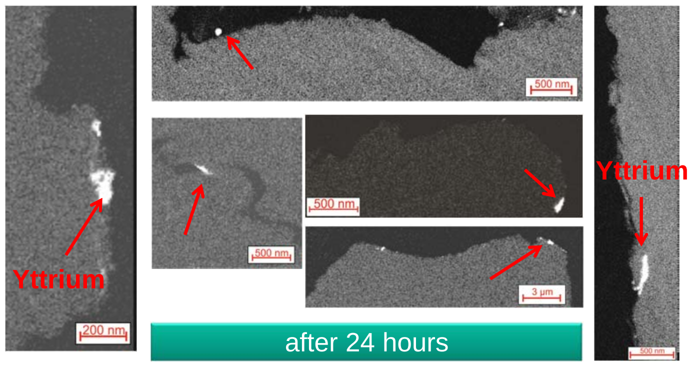
A benefit from longer milling-times is the homogenization of particle-size-distribution, which leads to a more dense compacted material after HIPping.

Discussion

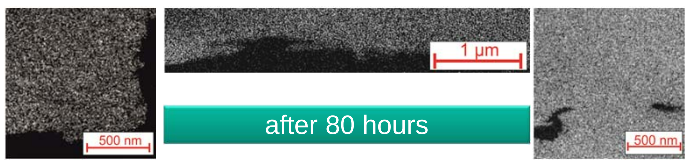
The thin slices performed very well for HAADF and EDX mappings. A STEM image of a powder particle is shown here:



A non-homogenous distribution of Yttrium could be found in the specimen.
The fact, that Yttrium could be very well detected in the specimen after 24h milling-time, leads to the conclusion, that the solution during the MA was not finished.



After longer milling times (48h, 80h) the Yttrium-rich region disappeared and couldn't be detected anymore.



Conclusion and Outlook

TEM sample preparation for MA steel powders by a microtom, proved to be a fast method for the validation of the milling time. Small batches of a new material for different times and examined using TEM + EDX. Then, the mechanical alloying process can be checked and validated without going through the complete ODS production process, which is highly time consuming and expensive.

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