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Synchrotron diffraction of nanostructured magnesium alloy WE43 prepared via cryomilling and spark plasma sintering

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１． Summary

The intermetallic phases present in the magnesium matrix were studied by synchrotron x-ray diffraction (XRD). The obtained data has given additional information that traditional XRD could not, which will provide insight into what other phases are present in this material. In addition to phase identification, this data has also given us a precise measurement of the lattice parameters of magnesium for all the samples which shows that the lattice parameters only vary slightly.

２． Purpose of experiment and background

Magnesium (Mg) alloys have long been an area of research due to the low density and high strength-to-weight ratio, but have yet to reach widespread adoption due its relatively low strength compared to other alloy systems such as aluminum. However, recent improvements in strength and creep resistance have been achieved with the addition of yttrium (Y) and rare earth (RE) elements such as neodymium (Nd) and gadolinium (Gd). One of the most popular of these commercial alloys is WE43 (4 wt. % Y, 3 wt. % RE, Mg balance) which achieves its maximum strength from traditional casting with the additional of a T6 temper (525 ºC for 8 hrs solid solution treatment + water quench + 250 ºC for 16 hrs aging). The majority of the improved strength and creep resistance compared to traditional aluminum-based magnesium alloys is attributed to precipitation hardening from Mg-RE intermetallic compounds. Due to the casting process and subsequent solution treatment, small grains are not achieved in this material and therefore this material traditionally does not see any enhanced mechanical properties due to grain refinement.

Our current research takes this WE43 alloy and applies special processing to achieve nanocrystalline grains in order to achieve additional strength improvements. This process involves taking WE43 gas atomized powders (“**as-received powder**”) and cryomilling (mechanical ball billing in liquid nitrogen) for 8 hours

which results in a powder with a final grain size of 20 nm ("**cryomilled powder**"). These nanocrystalline powders are then consolidated in a process known as spark plasma sintering (SPS) which applies pressure (100 MPa) and heat (400 ºC) in the form of a pulsed DC current for ~5 min in order to achieve a bulk material ("**as-SPS'ed**") while retaining most of the nanocrystalline structure.

However, due to the nanocrystalline nature of this material and unique processing route, it is not possible to characterize the phases present in the cryomilled powder and as-SPS'ed bulk sample by traditional means. It is unclear in what way the precipitation formation/sequence of the intermetallic compounds differs from the traditional WE43 aging process and in what way the large grain boundary volume fraction influences this. If a large majority of the RE elements are segregated to the grain boundaries, this could affect what precipitates can form and in what relative quantities.

The aim of this study is to use synchrotron x-ray diffraction to identify the intermetallic phases present in the alloy as well as to measure the lattice parameter of the magnesium matrix. Identifying the intermetallic phases of the precursor powders (as-received and cryomilled) will show the effect of the mechanical processing before consolidation and whether or not any intermetallic compounds are present before consolidation. It is hypothesized that the cryomilling process pushes the RE elements into a super saturated solid solution due or forces them into the high volume of grain boundaries and therefore does not contain any intermetallic compounds. Measuring the lattice parameter will help determine where the RE elements are located if not located in intermetallic compounds.

Additionally, identifying the intermetallic phases and measuring the lattice parameter of the magnesium matrix after consolidation (as-SPS'ed) and subsequent heat treatment will show what intermetallic compounds are formed and how stable these compounds are during post-consolidation heat treatments. This will also provide information of the aging response of the material (if any) as well as the thermal stability. With this information, we will be better able to predict the mechanical deformation mechanisms as well as routes for future improvements to the mechanical properties of future alloy developments.

3. Experimental

The XRD experiments were carried out using a standard diffractometer installed at BL07. The experiments will be conducted at incident x-rays energies of 25 keV at room temperature. Debye-Scherrer rings will be recorded on the 2D detector (PILATUS 100K) with an area of 487 × 195 pixels with a pixel size of 172 × 172 μm^2.

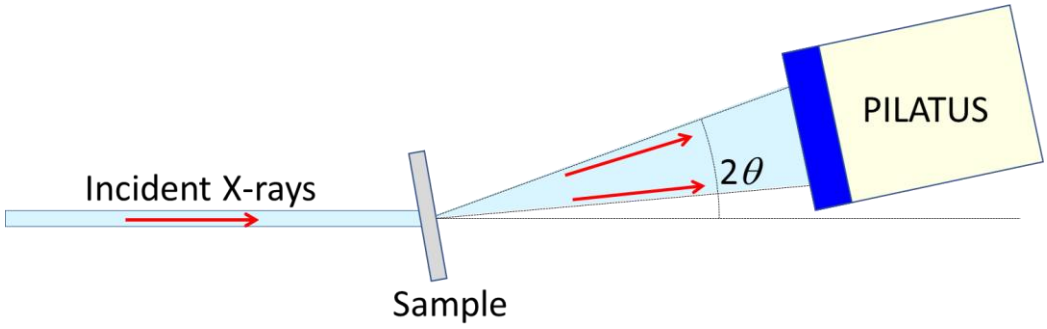

Fig 1: Schematic setup for the XRD experiment. The 2D detector (PILATUS) was used to collect the diffraction intensity in a 2θ range.

4. Results and Discussions

The data acquired was for the as-received powders (Sample 01), the cryomilled powders (Sample 02), the

as-SPS'ed sample or as-compacted/bulk sample (Sample 03), and four heat treatments of the as-SPS'ed sample (Samples 04-07). All samples show six characteristic diffraction peaks from Mg, and two characteristic diffraction peaks from magnesium oxide (MgO) with the exception of Sample 01 which does not contain the oxide. Sample 01 shows the presence of an intermetallic containing Mg and rare earth (RE) elements (Fig. 2). Besides these three phases just mentioned, the other phases (below 10 degrees) have yet to be identified. These phases do not show up in traditional XRD (Cu K-alpha radiation) that we have run, so it is still unclear what they could be. Possible candidates for these phases include other Mg-RE intermetallic compounds, intermetallic compounds not containing Mg, or impurities from the cryomilling process. It is interesting to note that Sample 02 contains a phase(s) which is/are not detected in any of the other samples as shown in Fig. 3. Additionally, there seems to be a phase (around 5 degrees) that is present in all of the samples.

In addition to phase identification, this data has also given us a precise measurement of the lattice parameters of magnesium for all the samples which shows that the lattice parameters only vary slightly. We also can see that this material is thermally stable with peak intensities not changing, with the exception of Sample 07 which shows the first MgO peak slightly collapsing which most likely is a result of this phase coalescing into larger grains. However, despite this, the samples show remarkable thermal stability.

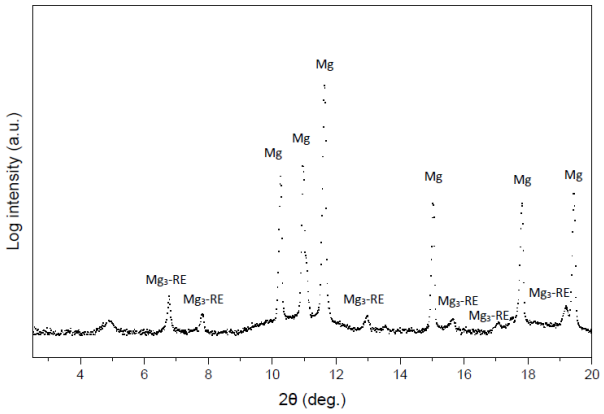

Fig 2: XRD pattern for Sample 01.

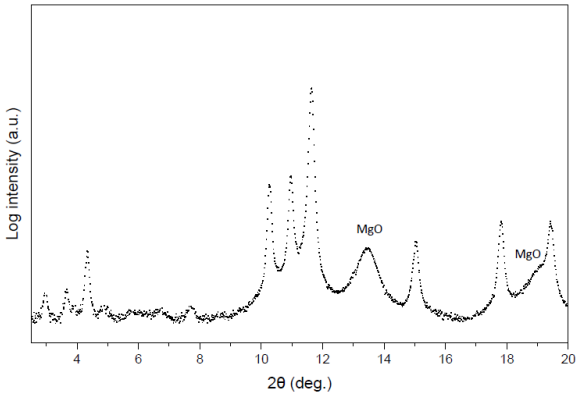

Fig 3: XRD pattern for Sample 02.

5. Future issues

This synchrotron data has given additional information that traditional XRD could not, which will provide insight into what other phases are present in this material. Additional analysis of the XRD diffraction results will need to be performed to reveal which phases these are, as there is limited information on such phases.

６．References

７．Publications, patents

８．Keywords

Magnesium matrix, XRD, Nanocrystallin

９．About the publication of research results

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