# Experiment report on ESRF – HS-2968

## *PDF from high $Q_{max}$ powder diffraction to monitor the local atomic structure changes in β-$Al_3Mg_2$ by plastic deformation*

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In technologically interesting lightweight alloys Al-Mg ($\rho \approx 2.2$ gcm$^{-1}$), a phase β is present around 60at% Al. The crystal structure of β-$Al_3Mg_2$, cF1832-664, *Fd*-3*m*, $a$ = 28.34 Å was solved and described in 1964 by Samson [1]. The structure contains 23 different atomic orbits, 11 (!) of which are not fully occupied. It represents one of the most complex metallic alloy (CMA) in terms of unit cell dimensions and atomic disorder. Although known since 40 years by now, the nature of the atomic disorder is not well understood today [2]. Moreover, β-$Al_3Mg_2$ shows an interesting plastic deformation behaviour; deformed samples so far have been characterized by TEM [3].
Now, three samples of polycrystalline β-$Al_3Mg_2$ have been investigated in experiment HS-2968 by high resolution powder diffraction at ESRF-ID31:

| **sample** | **sample history** |
|---|---|
| FAM2x | as grown (Bridgman) |
| FAM224ref (≡ FAM2_24ref) | non-deformed part of FAM2_24, heated to 350°C |
| FAM224slip (≡ FAM2_24verf) | uniaxially deformed part (slip plane) of FAM2_24: strain rate $10^{-4}$s$^{-1}$ at 350°C, strain ca. 6% |

**Experimental:**
The samples were cooled in liquid nitrogen and powdered at 80K in an agate mortar. The powders were placed in glass capillaries of 0.8mm diameter and continuous 2θ scans were performed in approx. 1h from 2θ = 0 to 120° at uniform filling mode of the storage ring ($I_{max}$ = 200mA). Eight scans were averaged for each sample (bin sizes 0.001° and 0.003°). The wavelength selected was λ = 0.35077(1)Å. The highest usable diffraction angle was 2θ = 112° which corresponds to $Q_{max}$ = 29.7Å$^{-1}$. Two scans of an empty capillary were averaged and smoothed to obtain the instrumental background.

To extract the total scattering [4], the smoothed background was subtracted and the total scattering function had to be corrected empirically for an instrument effect, assuming that the total scattering curve follows the average atomic scattering factor for high diffraction angles (Fig. 1).

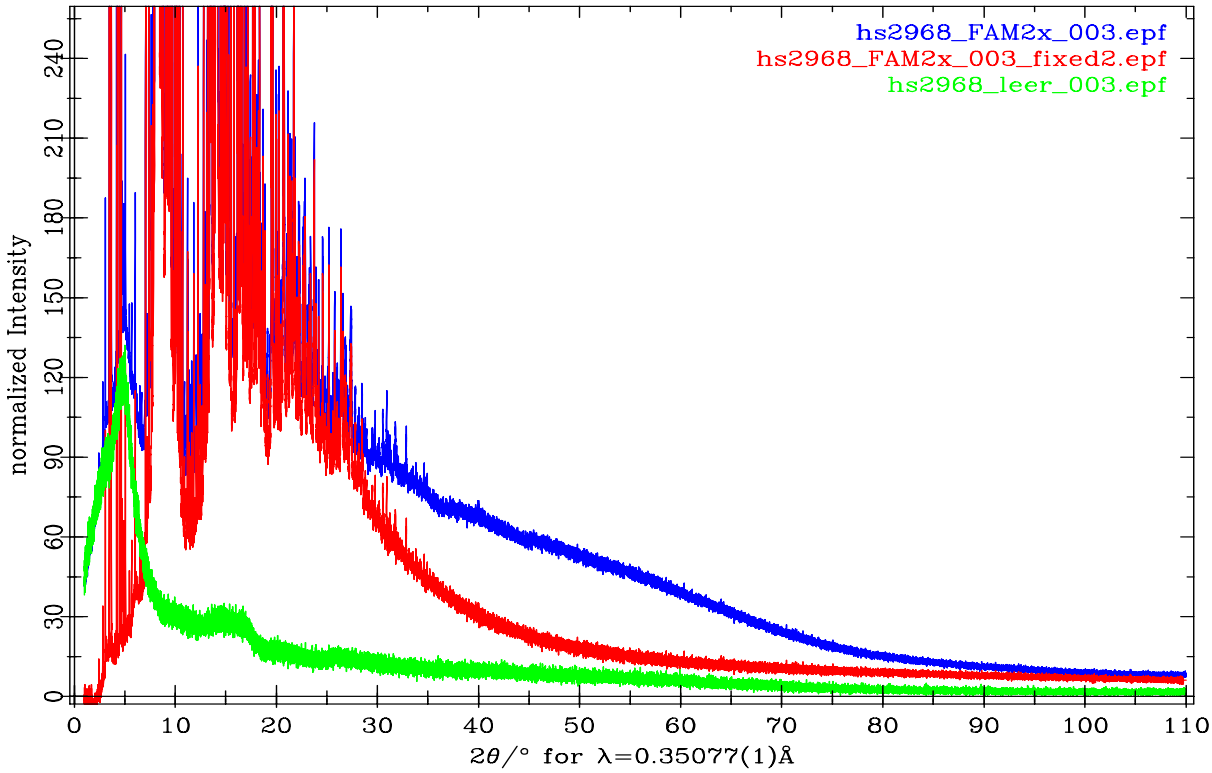


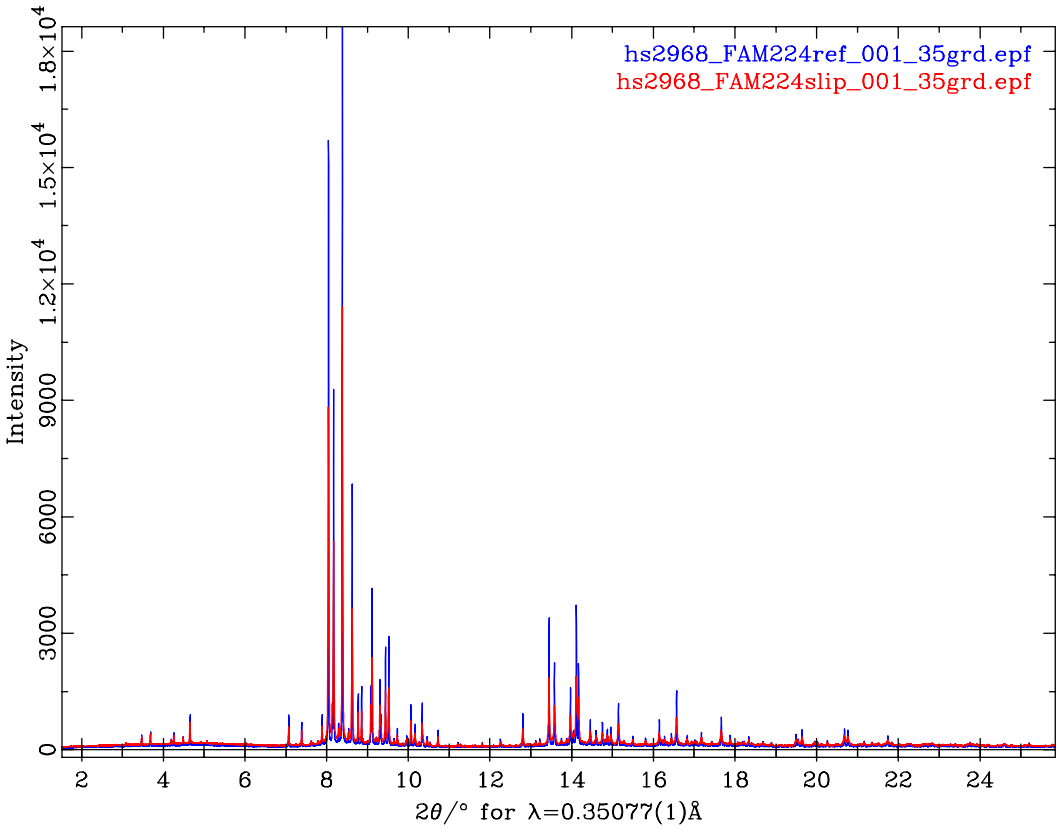

Fig 1. Total scattering of FAM2x, normalized Intensity $I(2\theta)$: blue: as measured (average of 8 scans), green: experimental background (average of 2 scans), red: corrected for background and instrument effect (~0.1%; note: $I_{max}$ = 17,900)

Fig. 2 shows low angle portions of the high resolution diffractograms, corrected for background and instrument effects. Blue: sample FAM224ref; red: deformed part (slip plane) FAM224slip. Note the eminent broadening of the reflection profiles: The mechanical deformation causes a larger FWHM (factor 3 to 4) of the reflections around the strongest Bragg reflection ($2\theta$ = 8 to 9°, Fig. 2b). Reflection positions are left unchanged.

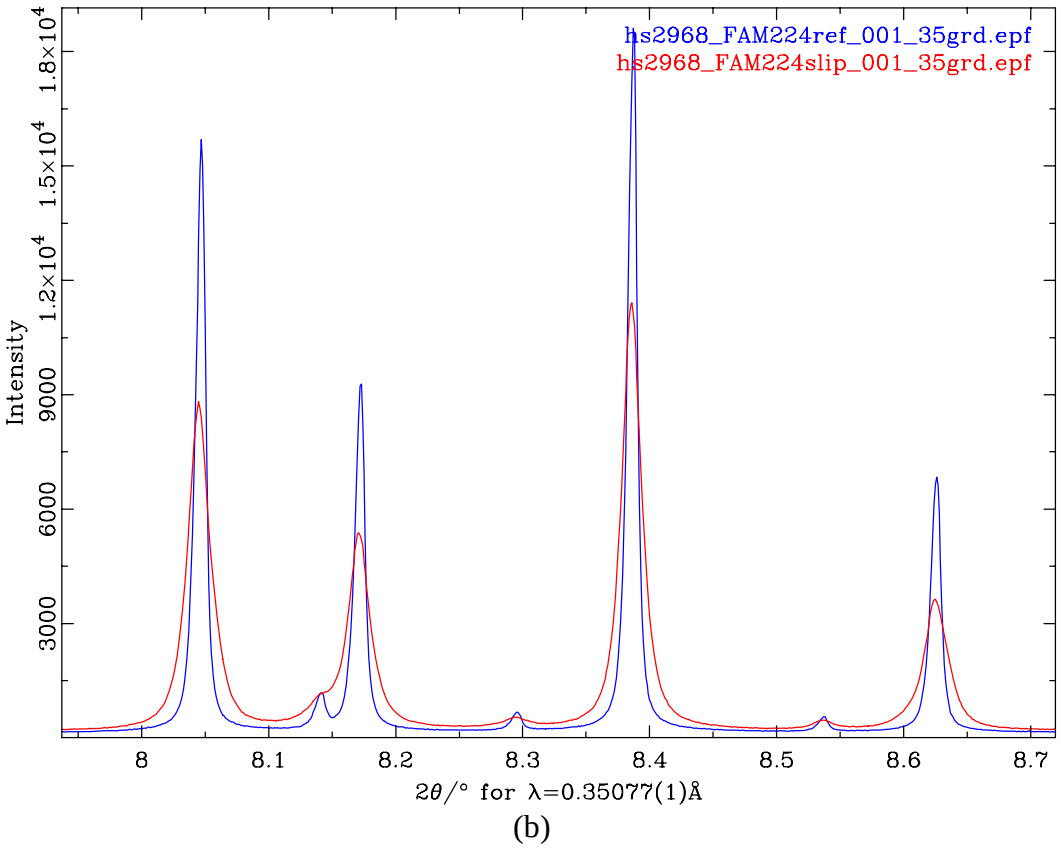
The reduced structure functions $F(Q) = Q[S(Q)-1]$ and the atomic pair distribution functions (PDF) $G(r)$ were obtained using the program PDFgetX2 [5] and are displayed in Fig. 3. Choosing different $Q_{max}$ for the Fourier transform, it turns out that $Q_{max}$ = 18Å$^{-1}$ yields the best compromise in between PDF resolution and the absence of truncation effects. While FAM2x and FAM224ref compare well in maximum $F(Q)$ and noise at high $Q$, there is a significant increase of noise for $Q$ > 12Å$^{-1}$ for deformed FAM224slip. This is accompanied by decrease of Bragg peak height due to reflection broadening (compare Fig. 2b).

Accordingly, the PDFs of FAM224ref and FAM224slip differ as is evident from Fig. 4. Here a $Q_{max}$= 12Å$^{-1}$ was chosen to lower PDF resolution stressing the main features of the PDFs up to $r$ = 100Å.

Fig. 5 shows $F(Q)$ and the PDF for FAM2x recently obtained by neutron diffraction at the NPDF instrument at Los Alamos National Laboratory, M. Lujan Neutron Scattering Center LANSCE-12. The X-ray experiment HS-2968 at ESRF now complements these data for the sample FAM2x: compare to Figs. 3a and d.




(a)




(b)

Fig.2 Low angle portion of the corrected diffractograms of FAM224ref and FAM224slip

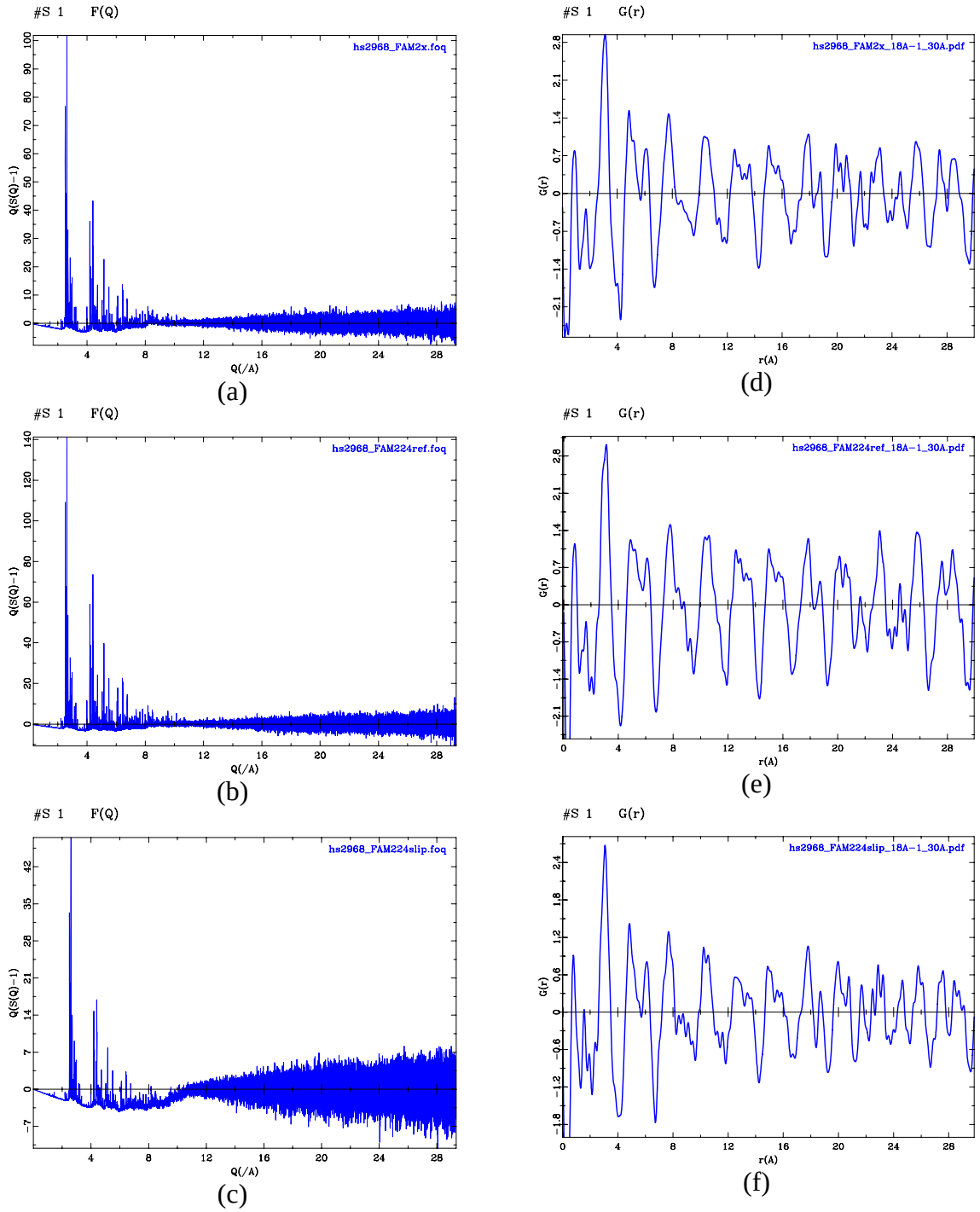
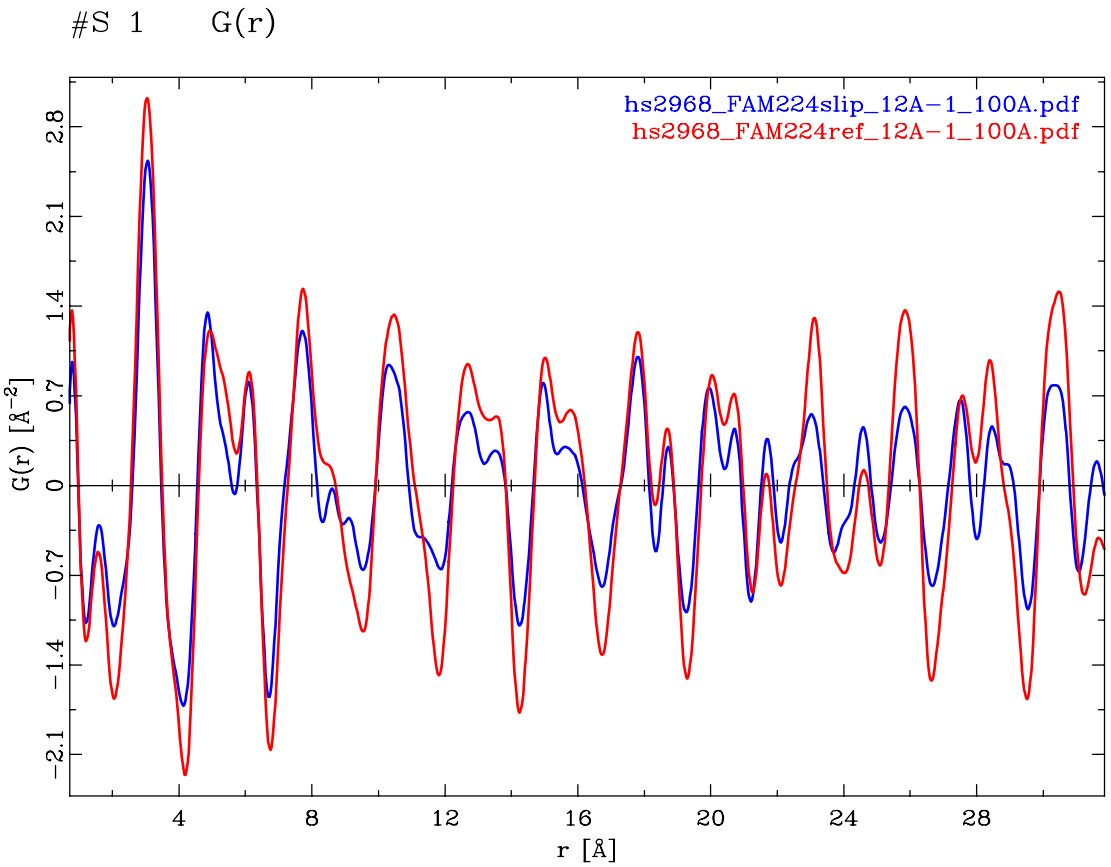
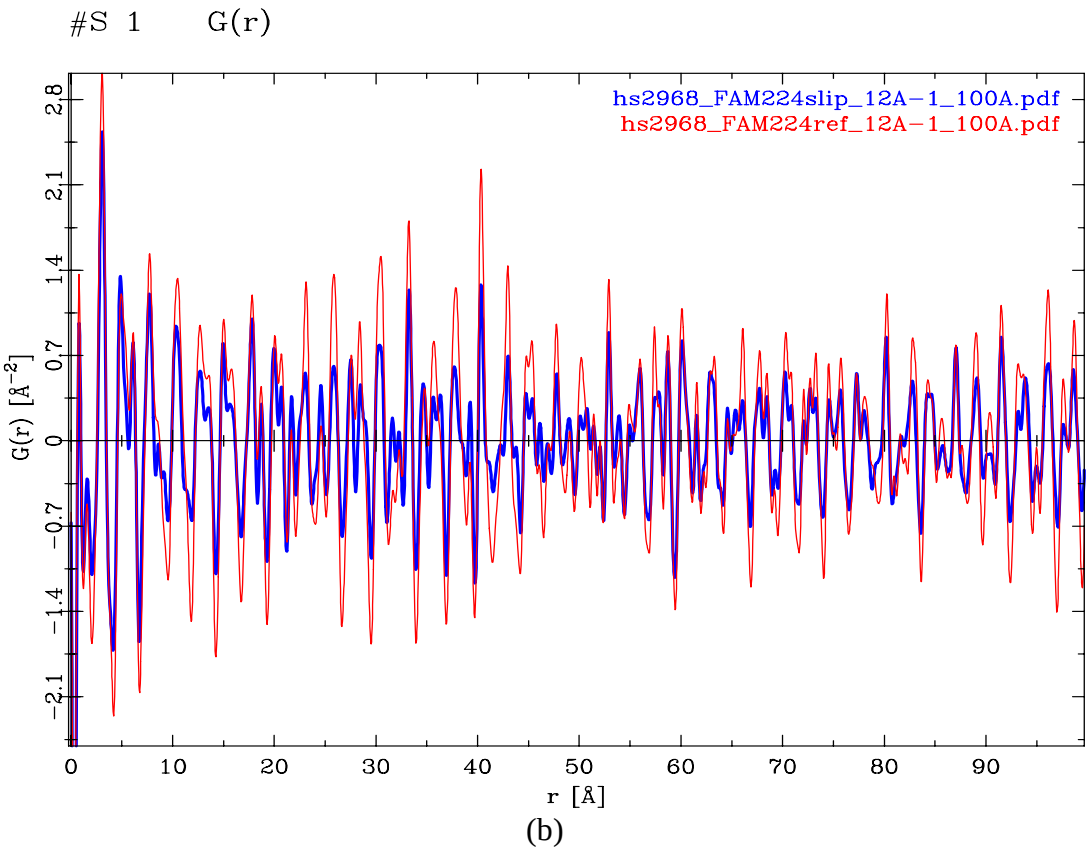



Fig. 3 $F(Q)$ and $G(r)$ ($Q_{max} = 18A^{-1}$) for FAM2x (a,d), FAM224ref (b,e) and FAM224slip (c,f)

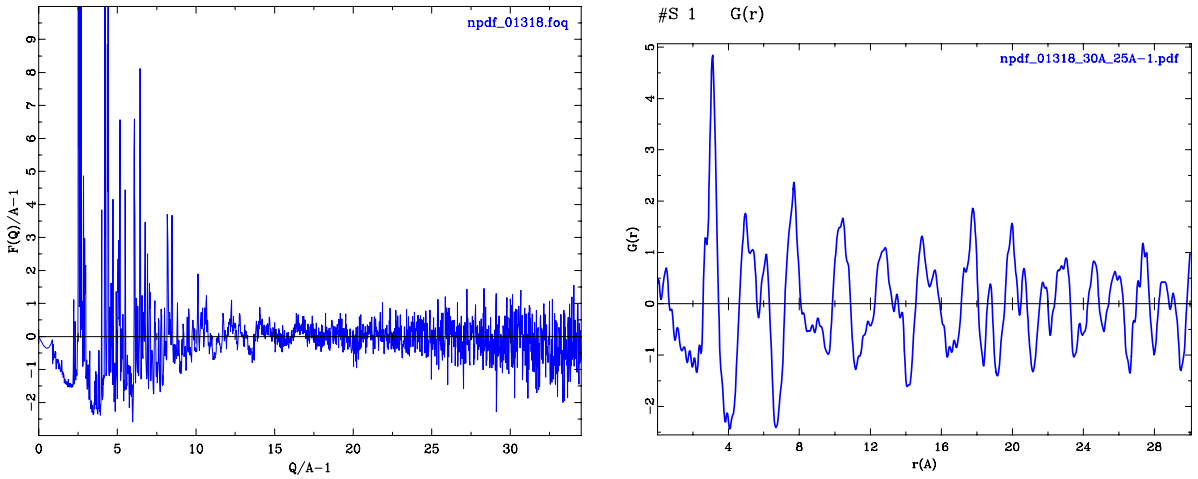



(a)




(b)

Fig. 4 Comparison of the PDFs ($Q_{max}$ = 12A$^{-1}$) of the deformed (red: FAM224slip) and non-deformed (blue: FAM224ref) parts of β-$Al_3Mg_2$ up to (a) $r$ = 3nm and (b) $r$ = 10nm.




(a) (b)

Fig. 5 Neutron powder diffraction of FAM2x. (a) Reduced structure function $F(Q)$, (b) PDF $G(r)$ ($Q_{max}$ = 25A$^{-1}$). The experiment (npdf_01318) was conducted at the NPDF instrument at Los Alamos National Laboratory, M. Lujan Neutron Scattering Center LANSCE-12, Los Alamos, USA

**Results**

- High-resolution synchrotron X-ray powder diffraction data up to $Q_{max} \approx 30$A$^{-1}$of β-$Al_3Mg_2$, both as grown and mechanically deformed, are now available.

- The samples are phase pure; deformed parts show a significant broadening of the Bragg reflections, different high-$Q$ scattering and thus differences in the corresponding PDFs.

- For the as grown material, X-ray data are complemented by neutron diffraction data.

-The data therefore suit subsequent*)

- reciprocal space Rietveld refinements and
- real space PDF local structure refinements

| β-$Al_3Mg_2$ | | as grown | deformed |
|---|---|---|---|
| Rietveld | X-ray | yes | yes |
| | neutron | yes | no |
| PDFFIT | X-ray | yes | yes |
| | neutron | yes | no |

*) The data will be used in WP6 and WP17 of the joint structure investigation activities within the FP6 EC-NoE “Complex Metallic Alloys” (CMA; www.cma-ecnoe.org). They will be complemented by X-ray and neutron single crystal diffraction data.

**References**

[1] Samson S 1964 *Acta Crystallogr.* **19** 401
[2] Urban K & Feuerbacher M 2004 *J. Non-Cryst. Solids* **334&335** 143
[3] Feuerbacher M, Heggen M & Urban K 2004 *Mat. Sci. Eng.* **375-377** 84
[4] Egami, T & Billinge S J L, 2003 *Underneath the Bragg Peaks: Structural Analysis of Complex Materials*, Pergamon Press Elsevier, Oxford, England
[5] Qiu, X, Thompson, JW; Billinge, SJL 2004 *J. Appl. Cryst* **37** 678