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J M Dubois, P. Chieux, Gérard Le Caër, J. Schweitzer, J. Bletry. DETERMINATION BY PO-LARIZED NEUTRON DIFFRACTION OF THE THREE PARTIAL STRUCTURE FACTORS OF A Co81.511B18.5 GLASS. Journal de Physique Colloques, 1982, 43 (C9), pp.C9-23-C9-29. 10.1051/jphyscol:1982904. jpa-00222397

### HAL Id: jpa-00222397 https://hal.science/jpa-00222397

Submitted on 4 Feb 2008

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# DETERMINATION BY POLARIZED NEUTRON DIFFRACTION OF THE THREE PARTIAL STRUCTURE FACTORS OF A CO81.5<sup>11</sup>B18.5 GLASS

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<u>Résumé</u> - Les trois facteurs de structure partiels (FSP) d'un alliage  $\operatorname{Co}_{81,5}^{11}B_{18,5}$ amorphes ont été mesurés simultanément par diffraction de neutrons polarisés. Un prépic de surstructure apparaît vers Q = 2 Å<sup>-1</sup> sur le facteur de structure nucléaire. Le FSP concentration-concentration montre que les atomes de bore sont uniquement entourés d'atomes de cobalt. On déduit les distances entre premiers voisins suivantes : R(Co-Co) = 2,54 Å, R(Co-B) = 2,3 Å, R(B-B)  $\sim$  3,6 Å. Enfin, les FSP calculés pour le modèle de GASKELL sont en accord satisfaisant avec les mesures tandis qu'apparaît un sensible désaccord entre l'expérience et le FSP S<sub>CC</sub> calculé pour un empilement de sphères adhésives.

<u>Abstract</u> - The three partial structure factors (PSF) of a Co $_{81,5}^{11}$ B<sub>18.5</sub> glass were simultaneously measured by polarized neutron diffraction. A appears around Q = 2 Å<sup>-1</sup> on the nuclear structure factor. The concentration-concentration PSF shows that the boron atoms are only surrounded by cobalt atoms. The following pair distances are deduced from PSF : R(Co-Co) = 2.54 Å, R(Co-B) = 2.3 Å, R(B-B)  $\sim$  3.6 Å. Finally the PSF calculated for the model of GASKELL are in satisfactory agreement with the measurements while a slight discrepancy is observed between experiment and the S<sub>CC</sub> FSP calculated for a packing of sticky spheres.

#### 1. Introduction

The diffraction of polarized neutrons is a unique way to measure in a single experimental run the three interference functions of a ferromagnetic binary M-X glass (1). In the usual experimental case, the neutron beam is polarized perpendicular to the scattering vector Q ( $|Q| = \frac{4\pi}{\lambda} \sin\theta$ ) and the electronic spins are aligned with the help of an external magnetic  $\lambda$  field H parallel (H // Q) or perpendicular (H  $\perp$  Q) to the scattering vector. Without polarization analysis of the scattered beam, the coherent scattering cross sections corresponding to the two  $\pm$  spin states of the neutron in both configurations are given by :

$$\frac{d\sigma}{d\Omega} \stackrel{\dagger}{=} (\bar{b} \stackrel{\dagger}{=} \bar{p})^{2} S_{NN}(Q) + 2(\bar{b} \stackrel{\dagger}{=} \bar{p}) \left[ (b_{M} \stackrel{\dagger}{=} p_{M}) - (b_{X} \stackrel{\dagger}{=} p_{X}) \right] C_{X} S_{NC}(Q)$$

$$+ \left[ (b_{M} \stackrel{\dagger}{=} p_{M}) - (b_{X} \stackrel{\dagger}{=} p_{X}) \right]^{2} C_{M} C_{X} S_{CC}(Q)$$
(1)

and

$$\frac{d\sigma}{d\Omega} = \frac{d\sigma}{d\Omega} = \overline{b}^2 S_{NN}(Q) + 2C_M \overline{b} b_M S_{NC}(Q) + C_M C_X (b_M - b_X)^2 S_{CC}(Q)$$
(2)

#### Article published online by EDP Sciences and available at http://dx.doi.org/10.1051/jphyscol:1982904

The symbols b and p represent respectively the nuclear and magnetic lengths,  $\mathbf{p}_{M}$  being related to the electronic magnetic moment  $\boldsymbol{\mu}_{M}$  located on element M by :

$$P_{\rm M}(Q) = 0.27 \ 10^{-12} \ F_{\rm M}(Q) \ \mu_{\rm M} \ (cm)$$
 (3)

where  $F_M(Q)$  is the normalized magnetic form factor of M.  $C_M$  and  $C_X$  are the atomic concentrations of elements M and X and

 $\bar{b} = C_M b_M + C_X b_X \cdot S_{NN}$ ,  $S_{NC}$  and  $S_{CC}$  are the BHATIA-THORNTON (2) partial structure factors (PSF) defined as :

$$S_{NN} = 1 + \rho \int \left[ C_{M}^{2} P_{MM} + C_{X}^{2} P_{XX} + 2 C_{M} C_{X} P_{MX} - 1 \right] e^{iQr} d_{3}r$$
  

$$density-density PSF$$

$$S_{NC} = \rho C_{X} \int \left[ C_{M} P_{MM} - C_{X} P_{XX} + (C_{X} - C_{M}) P_{MX} \right] e^{iQr} d_{3}r$$

$$density-concentration PSF$$

$$S_{CC} = 1 + \rho C_{M} C_{X} \int \left[ P_{MM} + P_{XX} - 2 P_{MX} \right] e^{iQr} d_{3}r$$

$$concentration-concentration PSF$$

$$(4)$$

 $\rho$  is the atomic density and  $P_{\alpha\beta}(r)$  the probability per unit volume (normalized to unity at large values of r) to find the  $\alpha$  species at a distance r of a  $\beta$  atom. The BHATIA-THORNTON PSF are linearly related to the FABER-ZIMAN (3) PSF which will also be used in this study.

be used in this study. The difference  $\frac{d\sigma}{d\Omega} \perp - \frac{d\sigma}{d\Omega} \perp$  is proportionnal to the magnetic form factor F(Q) of the alloy. Therefore, the accuracy of the method is related to the Q dependence of F(Q) and vanishes when F(Q) becomes too small. For this reason, the useful Q range is in fact limited to  $Q \leq 7 \text{ Å}^{-1}$ . This is a serious restriction on the informations available from the method, especially regarding the Fourier transforming of the structure factors. However, the diffraction of polarized neutrons is a unique way to solve the structure of alloys which are not the concern of the isotopic substitution method. This is the case of the Co-B alloys because cobalt has only one stable isotope (<sup>59</sup>Co) and because the use of <sup>10</sup>B, one of the two boron isotopes, is ruled out due to its too large absorption cross section.

The purpose of this paper is to report on the measurement of the three PSF of a  $\operatorname{Co}_{81,5}^{11}B_{18,5}$  glass. The results are compared to data concerning similar transition metal-metalloid glasses. These PSF are also used to check the models of GASKELL (4) and of BLETRY (1,5).

#### 2. Experimental

The master alloy was prepared by sintering the pure (99.9 %) elements in powder under secondary vacuum in a sealed quartz tube. Boron 97.2 % enriched in <sup>11</sup>B was used. The glass was produced in ribbon shape by melt spinning on a copper disc. The casting was made under an atmosphere of helium containing 5 % of hydrogene which avoids the reaction of cobalt with the quartz crucible. This process introduced a small amount of hydrogen in the glass which was measured out by chemical analysis to be 1.8 at. %. The sample weight was 8 g made of 50 mm long ribbon pieces mounted on an aluminium frame in a similar way as described by COWLAM et al. (6). A boron nitride shield protected this frame from the incident beam so that the irradiated volume remained constant with the scattering angle.

The experiment was performed with the D5 diffractomer of ILL (Grenoble) with a wavelength  $\lambda = 0.84$  Å. The neutron beam was polarized with the help of a Cu\_MnAl monochromator (polarization rate P = ~ 0.964). The spin state was changed every second by a flipping coil of efficiency  $\varepsilon = 0.925$ . Finally, the  $\lambda/2$  contamination of the beam was reduced to less than 0.5 % by a 0.75 mm thick erbium filter placed in front of the <sup>3</sup>He detector.

The electronic moments were aligned with an external field of 15 kG. In the  $(H \perp Q)$  configuration, the  $0.5 \leq Q \leq 12$  Å<sup>-1</sup> range was explored but the size of the polar pieces of the magnet limited the available Q range to  $0.5 \leq Q \leq 6$  Å<sup>-1</sup> in the  $(H \parallel Q)$  configuration. Despite this drawback, we have not changed the wave length in order to keep the optimal flipperefficiency and the same sampling step as used in the  $(H \mid Q)$  experiment.

#### 3. Results

The correction of the rough intensities for absorption, background and beam polarization was performed as already described by BLETRY (1). These corrected intensities are shown on figure 1.

For Q  $\leq$  0.6 Å<sup>-1</sup>, the (H  $\int$  Q) intensities exhibit a diffuse scattering component



which is not visible on the nuclear interference function  $(H/\!/Q)$ . This component is therefore of magnetic nature but its origin remains unknown.

The incoherent cross section of hydrogen is one order of magnitude larger than that of cobalt and drops rapidly with the diffraction angle. Therefore, the contribution of a small amount of this element cannot be neglected as is demonstrated by the decrease at large Q values of the intensities of figure 1. Before normalizing the data, we have thus corrected the intensities with the help of empirical relations developed by CHIEUX in a previous study (P. CHIEUX, B. BOUCHER, to be published) :

$$\frac{d\sigma}{d\Omega} \frac{inc}{d\Omega} (2\theta) = A \cos 2\theta \qquad 2\theta \le 2\theta_{0}$$

$$\frac{d\sigma}{d\Omega} \frac{iH}{nc} (2\theta) = A \frac{\theta_{m} - \theta}{\theta_{m} - \theta_{0}} \cos 2\theta_{0} \qquad 2\theta > 2\theta_{0}$$
(5)

with  $2\theta_m = 110^\circ$ ,  $2\theta_p = 45^\circ$  and A proportionnal to the hydrogen content.

The corrected intensities were then proportionnal to the total cross sections of the sample without hydrogen. The proportionnality constant was calculated (1) at large values of Q where nuclear and magnetic interferences have vanished. This requires to expand the (H//Q) pattern in the  $6 \le Q \le 12$  Å<sup>-1</sup> range. Taking into account that  $F(Q) \Rightarrow 0$  when Q > 6 Å<sup>-1</sup>,  $d\sigma///d\Omega$  can be written as

$$\frac{\mathrm{d}\sigma}{\mathrm{d}\Omega} = \frac{\mathrm{R}}{2} \left[ \frac{\mathrm{d}\sigma}{\mathrm{d}\Omega} + \frac{\mathrm{d}\sigma}{\mathrm{d}\Omega} \right] \qquad \mathrm{Q} > 6 \mathrm{A}^{-1}$$

where R accounts for an eventual variation of the neutron flux when changing the experimental configuration and  $X \leq 1$  averages the actual small but non zero values of x(x)

F(Q). Finally, the cross sections were corrected for the nuclear incoherent, inelastic (7) and elastic multiple (8) scattering of Co and B. As shown by the NMR study of Co-B glasses (9), the standard deviation of the magnetic moment distribution is small ( $\Delta p^2(0) \leq 0.05 \ p^2(0)$ ) and the measurement temperature being far below the temperature, the incoherent magnetic scattering was

neglected. Representative values of the different cross sections are reported in table 1 and the normalized coherent cross sections on figure 2.

The total nuclear structure factor  $\frac{d\sigma}{d\Omega}^{\prime\prime}$  exhibits a strong prepeak around  $Q = 2 \stackrel{A^{-1}}{A^{-1}}$  and a well resolved shoulder of the second intense peak which amounts near 1. Both features are also characteristics of the neutron diffraction pattern published for the same glass by LAMPARTER et al. (10). On the other hand, no prepeak is visible on the pattern of CHADHA et al. (11) which seems to have been arbitrarily cancelled out for  $Q \leq 2.5 \stackrel{A^{-1}}{A^{-1}}$  and does not exhibit a marked shoulder.

Q Å-1	$4\pi \overline{b}^{2}$ $10^{-24} \text{ cm}^{2}$	$\begin{array}{c} \text{Co-B} \\ \text{Oinc} \\ 10^{-2.4} \text{ cm}^2 \end{array}$	$\frac{H}{\sigma_{inc}}$ 10 <sup>-24</sup> cm <sup>2</sup>	$\frac{\sigma_{mu1}}{10^{-2} cm^2}$	<sup>0</sup> inel 10 <sup>-24</sup> cm <sup>2</sup>	F(Q)	$4\pi \ p^{2}$ $10^{-24} \ cm^{2}$
1	1.544	4.15	0.113	0.013	0.002	0.947	2.417
6	1.544	4.15	0,080	0.013	~ 0.007	0.215	0.125
12	1.544	4.15	0	0.013	- 0.034	0	0

Table 1



Figure 2 - Normalized coherent cross

sections of the Co<sub>81.5</sub><sup>11</sup>B<sub>18.5</sub> glass.

The normalized magnetic form factor of cobalt was defined according to LISHER and FORSYTH (13) as :

 $F(Q) = 0.3749 \exp(-0.00942 WQ^2)$ 

+ 0.6375 exp (- 0.03164 WQ<sup>2</sup>) - 0.0131 by introducing (1) a width parameter W which accounts for the actual extension of F(Q). Except in the forward direction were no measurement is made, the actual moment  $\mu_{Co}$  experienced by the scattered neutron is larger than the average moment  $\mu$  measured by the bulk magnetization, i.e. :

 $\mu_{CO} \approx \alpha \overline{\mu}$  with  $\alpha \ge 1$  and  $\overline{\mu} = 1.32 \mu_B/CO$  at. (13).

The parameters W and  $\alpha$  were ajusted until the PSF oscillated around 1 (S<sub>NN</sub> and S<sub>CC</sub>) or O(S<sub>NC</sub>) and satisfied the usual integral relations (1,2). The best couple of values was {W, $\alpha$ } = {0.95, 1.3} corresponding to a form factor which is not significantly different from that of metallic cobalt while the contribution to the magnetization of the conduction electron polarization, near - 0.4  $\mu_{\rm B}$ /Co at., is somewhat larger than that of metallic cobalt (- 0.28  $\mu_{\rm B}$ /Co at.).

The BATHIA-THORNTON PSF of the Co $^{11}$ B glass are reproduced on figure 3. Due 18.5 to the lack of (H // Q) measured intensities above Q = 6 A<sup>-1</sup>, they are only significant in the  $1 \leq Q \leq 6$  Å<sup>-1</sup> range.

As expected from the presence of a prepeak,  $\stackrel{S}{\text{--}}_{\text{--}} \stackrel{S}{\text{---}}_{\text{---}} \stackrel{S}{\text{----}}_{\text{----}}$  This peak is unusually broad. This indicates that the alloy is strongly ordered. From a comparison with the PSF calculated by BLETRY for models of dense packing of sticky spheres (1, 14) varying the COWLEY parameter (15), one deduces  $\xi \simeq -0.25$ . This value is

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close to  $\xi = -0.23$  expected from the composition for boron atoms only surrounded by cobalt atoms. The extrapolation of  $S_{CC}$  to Q = 0 leads to  $S_{CC}(0) \simeq 0.35$ . Assuming according to NOLD et al. (16), that the glass structure is close that of the undercooled melt during the glass transition,  $S_{CC}(0)$  yields the excess stability function  $E^{xs}$ . Taking Tg as the start of crystallization temperature  $T_{CR} \simeq 600$  K (17), one finds  $E^{xs} \sim 60$  kJ/mol which is also calculated under the above assumption for Fe<sub>30</sub>B<sub>20</sub> and Ti<sub>40</sub> Ni<sub>60</sub> glasses(16)It is also worth noticing the peak positions of our PSF are near those of Fe<sub>80</sub>B<sub>20</sub> (16).



Figure 3 - BHATIA-THORNTON partial structure factors of the Co<sub>81.5</sub><sup>11</sup>B<sub>18.5</sub> glass.

Figure 4 - FABER-ZIMAN partial structure factors calculated from the PSF of figure 3.

0

 $S_{NC}$  oscillates around zero because the atomic radii ratio is significantly different from 1 ( $r_{CO} = 1.25$  Å,  $r_{B} = 0.87$  Å) and  $S_{NN}$  shows a first intense peak at  $Q_{NN} = 3.15$  Å<sup>-1</sup> with  $S_{NN} = 2.7$ . The position of this peak agrees with the relation  $Q_{NN} = \pi \sqrt{3}/\sqrt{2}$  R<sup>-1</sup> = 2.26 Å (1,14) where R is the average atomic radius.

Finally, figure 4 presents the FABER-ZIMAN PSF calcuted from the preceeding PSF. The following mean pair distances  $R_{\alpha\beta}$  are deduced from the positions  $Q_{\alpha\beta}$  of the first maxima by using the phenomenological relation of BLETRY :

 $Q_{\alpha\beta} = \overline{R}^{-1} \left[ 7.6 - 4.3(2R \ \overline{R}_{\alpha\beta}^{-1} - 1) \right] (1, 14) \text{ which is similar to the well known relation of EHRENFEST (18) :}$ 

 $\overline{R}_{CoCo}$  = 2.54 <sup>±</sup> 0.03 Å,  $\overline{R}_{Co-B}$  = 2.3 <sup>±</sup> 0.1 Å,  $\overline{R}_{BB} \sim 3.6$  Å.

Despite the slight asymetry of  $S_{COCO}$ , it provides a satisfactory accuracy for the measurement of  $\overline{R}_{COCO}$  and both the asymetry of the peak and the distance are in excellent agreement with EXAFS measurements in the same sample :  $\overline{R}_{COCO} = 2.56 - 0.01 \text{ Å}$  (19). The strong dissymetries of the  $S_{CO-B}$  and  $S_{B-B}$  peaks avoid determining precise pair distances which are however similar to the distances published elsewhere for this glass (10,11) or known for the orthorhombic  $Co_3B$  boride (20).

#### 4. Comparison with models

In order to check the Trigonal Prismatic Packing (TPP)model of GASKELL (4), we compare on figure 5 our experimental PSF to the PSF calculated from the atomic positions of the model. The calculation was performed with the coordinates of the most inner atoms as described by GASKELL (4). However, as the model was built for  $Pd_{80}Si_{20}$ , we have reduced the coordinates according to the Co/Pd radii ratio without taking into

account the shape difference of the basic prismatic units encountered in the isomorphous  $Pd_3Si$  and  $Co_3B$  compounds. For this reason, the geometric PSF  $S_{NN}$  is not in quite satisfactory agreement with the experience though the positions of the peaks are correctly accounted for. On the countrary, the prediction of the model looks better for the two other PSF, especially for  $S_{CC}$  whose first peak width compares well to the experimental one.

A dense packing of 5000 sticky spheres was computer built for the composition  $M_{\rm go}X_{20}$ as already described in (1,5). The direct contacts between small spheres were forbidden in order to simulate the maximum chemical order. The spheres radii were taken equal respectively to the Co and B radii. No relaxation of the cluster was performed and the resulting density was 6.09 g cm<sup>-3</sup>. The amplitude of the PSF were thus corrected with respect to the experimental density d = 8.27 g cm<sup>-3</sup> (13).





The PSF calculated from the atomic posi- the model of GARAND. The PSF calculated from the experimental results in figure 6. A satisfactory agreement is observed between measured and calculated  $S_{NN}$  and  $S_{NC}$ . However, a slight difference of phase is observed between the experimental and Calculated PSF. On the other hand, the width of the experimental  $S_{CC}$  is larger than the calculated PSF while these two functions seem to behave out of phase after the first peak. However, the accurary on the experimental  $S_{CC}$  is always rather poor. Despite this argument, a difference may exist which we assign to the model algorithm which allows small spheres being almost in contact. In the TPP model, this is explicitely forbidden by the packing of structural units. Moreover, the connectivity between the units resembles that of the crystalline Pd\_Si (Co\_3B) counterpart, leading thus to the formation of highly dissymetric metal shells respectively surrounding a metalloïde or a metallic atom. This last feature is in fact experimentally deduced from an EXAFS study of Co-B glasses (19). A broadening of the first peak of  $S_{CC}$ , and to a less extent  $S_{NN}$ , is therefore expected from the TPP model as actually shown. On the other hand, the packing of structy spheres seems to give a better account of more symetric structures as those of liquid alloys.

#### 5. Conclusion

This work is, to our knowledge, the second application of the polarized neutron diffraction method to the study of a metallic glass structure. It emphasizes the usefulness of the technique to solve problems which are not relevant of the isotopic substitution method as for amorphous  $Co_{81.5}^{B}$  18.5. From the existence of a prepeak on the total structure factors and from 1.5 18.5 the height of the first peak of the concentration-concentration partial structure factor, it is concluded that the boron atoms are only surrounded by cobalt atoms. A satisfactory agreement is observed between the experimental PSF and those calculated from the model of GASKELL and from a model of dense random packing of spheres. However, details are in favour of models based on the packing of structural units. A general description of such models is proposed elsewhere at this conference (21).

#### Acknowledgements

The allocation of beam facilities by ILL (Grenoble) is acknowledged. We are grateful to Dr. P.H. GASKELL for providing us with the coordinates of his model and to Dr. C. TETE for his help during the sample preparation.



Figure 6 - Experimental (dots) and calculated (dark line) PSF from a model of dense random packing of sticky spheres.

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