ESRF	**Experiment title:** EXAFS at Cu and Zr K-edge of $Zr_{65}Cu_{27.5}Al_{7.5}$ metallic glasses	**Experiment number:** HS-223
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Report:

Even the phenomenon of glass transition has been extensively examined since many years, there is still a considerable lack in its understanding. Metallic glasses are very interesting for applications because of their processability and excellent mechanical properties [l]. A good knowledge of the phenomenon of glass transition is important for understanding the properties of these materials.
From theoretical point of view, the glass transition has been described using thermodynamical and mechanical instability concepts [2]. The structural relaxation during the heating of the metallic glass to the under-cooled liquid regime can help to understand the glass transition. The short range order has shown to be important for thermodynamic properties [3]. The close connection between structural and thermodynamic properties is the aim of our interest and several work has been done so far to understand these connections [4]. The aim of this study were twofold:
By monitoring the changes of the local environment of Zr and Cu in the metallic glass $Zr_{65}Cu_{27.5}Al_{7.5}$ we wanted to investigate the local structure in order
1) to understand the properties of metallic glasses and
2) to have a better understanding of structural changes during the glass transition.

The experiment was performed at BM 29, using the L'Aquila-Camerino high temperature oven [5] working under high vacuum. One of the main concerns for this experiment was the time window available for EXAFS experiments in the under-cooled liquid regime. Figure 1 shows the absorption coefficient of the metallic glass as a function of temperature for a fixed incident photon energy, which was choosen to be highly sensitive to the crystallization transition. This shows that the thermal state of the sample can be monitored using this single energy detection technique developed at BM 29 [6]. We showed that it is not possible to perform experiments in the undercooled liquid regime with standard EXAFS acquisition time, which are at least around 20 minutes per scan, specially close to the crystallisation temperature. This is shown by the insert of Figure 1, which represents the evolution of the absorption coefficient as a function of time with fixed photon energy and temperature = 422 ^{o}C. One the other side, we could show, that the high temperature state can be quenched to a temperature were the glass is thermodynamically stable over a sufficient time. The experiment were therefore carried out in the following way: The sample was heated to a given temperature (300 to 480°C) and then quenched down to around 100°C were the EXAFS scan was performed. This procedure was applied in the same way at the Cu and Zr K-edge. The maximal temperature was ramped through the glass transition temperature (around 380°C) and furtheron over the crystallization temperature (around 470°C). Taking the EXAFS spectra always at a fixed low temperature has the additional advantage to cancel out thermal Debye-Waller like influence on the data.

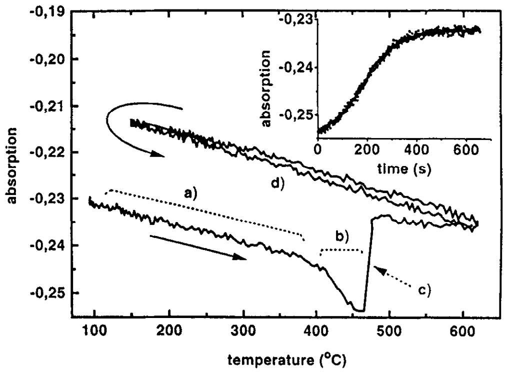

Figure 1): Single energy scan. a) Region of thermal relaxation and Debye-Waller effect, b) under-cooled liquid region, c) crystallization. The bold arrows indicate the sequence of the thermal treatment. The insert shows the evolution of the absorption coefficient at fixed T=422°C with time.

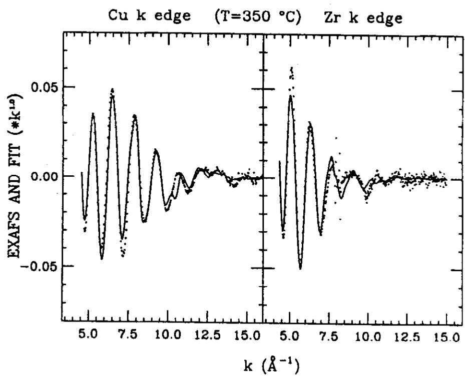

Figure 2): Simultaneous EXAFS analysis of Cu and Zr K-edge.

The spectra corresponding to the same maximal annealing temperature and thermal history taken on the Cu and Zr K-edge were analysed simultaneously using the GNXAS package [7]. Figure 2) shows an example for an annealing temperature of 350°C. We would like to underline that the analysis is not finished yet and that this are preliminary results. Nevertheless, the analysis starting with an amorphous model close to Zr_2Cu shows rather good agreement with other studies [8]. As a main results we only can detect minor changes in the coordination numbers and distances in the region of the under-cooled liquid. At the given state of the analysis, we conclude that the short range structure as seen via EXAFS remains basically the same in the region of interest with respect of the sensibility of EXAFS. A better starting model for the simulation is clearly needed to improove the data analysis and to be able to give more detailed and reliable information on the dynamics in the system.

References

[l] R. Bruening and K.Samwer, Physical Review B 46 (1992) 11318
[2] for review, see W.L. Johnson, Prog. Mater. Sci. 30 (1986) 81
[3] F. Sommer and D. K. Choi, Z. Metallkunde. 80 (1989) 263
[4] for example R. Rambousky, M. Moske and K. Samwer, Z. Phys. B 99 (1996) 387-391 M Weiss, M. Moske and KSamwer, to appear in Appl. Phys. Lett.
[5] A. Filipponi and A. Di Cicco, Nucl. Instr. and Methods 93 (1994) 302
[6] A. Filipponi, M. Borowski, P.W. Loeffen, S. De Panfilis, A. Di Cicco, F. Sperandini and M. Minicucci, Europ. J. Phys., Cond. Matter 10 (1998) 235-253
[7] A. Filipponi, A. Di Cicco, and C. R. Natoli, Phys. Rev. B 52 (1995) 15122 and 15135
[8] Bezzerra et al, J. Non. Cryst. Solids 126 (1990) 239