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Mössbauer effect and x-ray diffraction study of Zr–Ti–Cu–Ni–Be bulk metallic glasses

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Abstract

Phase separation and crystallization in the bulk amorphous alloys $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$ and $Zr_{46.7}Ti_{8.3}Cu_{7.5}Ni_{10}Be_{27.5}$ have been studied by Mössbauer spectroscopy and x-ray diffraction (XRD) investigations. Mössbauer spectra of Zr–Ti–Cu–Ni–Be with ^{57}Fe additions were measured during the course of isothermal annealing near their glass transition temperature. The spectra show a broad asymmetric quadrupole doublet which reveals the presence of different local environments. The values of the quadrupole splitting, centre shift and area ratios undergo characteristic changes in $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$ due to a decomposition process. An abrupt change of the parameters is observed when the sample begins to crystallize at 633 K after an annealing time of 600 min. This is in agreement with the results of XRD investigations that show sharpening of the diffraction maximum as a result of the formation of nanocrystals. The alloy $Zr_{46.7}Ti_{8.3}Cu_{7.5}Ni_{10}Be_{27.5}$ does not show any indications of decomposition and crystallization in the Mössbauer spectra. The processes are discussed from the viewpoint of the local atomic short-range order in the immediate neighbourhood of the Mössbauer atom.

1. Introduction

Many multi-component eutectic systems based on early transition metals exhibit excellent glass forming abilities and a high stability with respect to crystallization processes in the supercooled liquid above the glass transition temperature [1, 2]. The critical cooling rates to form these bulk metallic glasses from the melt are orders of magnitude lower compared to conventional metallic glasses. Their high thermal stability against crystallization has opened for the first time the opportunity to investigate thermodynamic and transport properties not only in the

solid state but also in the supercooled liquid state above the glass transition temperature [3, 4]. However, the thermal stability of the amorphous phase is finally limited by crystallization. In order to study the thermal stability, a detailed knowledge of the crystallization process is required. In the course of such studies uncommon pathways into the crystalline state have been observed in these glasses.

The crystallization behaviour of $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$ (V1) has been investigated by differential scanning calorimetry (DSC) [5], analytical field ion microscopy (APFIM) [6], transmission electron microscopy (TEM) [7] and small angle neutron scattering (SANS) [8]. These results show that primary crystallization is preceded by phase separation into two amorphous phases. SANS investigations reveal that predominantly Ti is involved in this decomposition process [9]. During decomposition, crystallization probability increases and finally a sharp crossover to a polymorph formation of nanocrystals occurs in the Ti rich domains.

In this respect, Mössbauer spectroscopy seems to be powerful, since it can provide useful information on the short-range atomic arrangement in the immediate neighbourhood of the Mössbauer atom. We therefore expect it to be of great value in understanding the nature of the decomposition and crystallization processes of the ZrTiCuNiBe bulk metallic glasses.

This paper is focusing on Mössbauer measurements of two series of Zr-based amorphous alloys $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$ (V1) and $Zr_{46.7}Ti_{8.3}Cu_{7.5}Ni_{10}Be_{27.5}$ (V4) where some Ni was substituted by the Mössbauer isotope ^{57}Fe . The characteristic Mössbauer spectra during the isothermal annealing programme were taken. Additionally, x-ray diffraction (XRD) investigations were performed to compare modifications which occur in the short-range atomic arrangement to changes on a scale of several nanometres during decomposition and crystallization.

2. Experimental details

Amorphous ZrTiCuNiBe samples were prepared from a mixture of the pure elements by induction melting on a water-cooled silver boat under Ti-gettered Ar atmosphere. The ingots were remelted in a silica tube with an inner diameter of 10 mm and then quenched with a cooling rate of about 10 K s^{-1} [7]. For the Mössbauer experiments, special ingots were prepared in which 1.5 at.% of the Ni atoms had been substituted by ^{57}Fe . Small amounts of about 20 mg were subsequently melted in a high frequency coil and quenched into thin foils with a twin-piston splat quencher under Ar atmosphere. These foils were used as absorbers.

The Mössbauer experiments were performed in transmission geometry at room temperature using a constant acceleration drive and a ^{57}Co (Rh) source. All isomer shifts IS were calibrated with respect to the α -Fe spectrum at room temperature. The V1 samples were annealed at 613 and 633 K, whereas the V4 samples were heat treated at 643 K for different times. For each individual annealing step a new virgin sample was used.

The microstructure of the samples was determined *ex situ* by XRD in Bragg–Brentano geometry using Cu $K\alpha$ radiation.

3. Results

3.1. Mössbauer spectroscopy

Room temperature transmission Mössbauer spectra of ZrTiCuNi(Fe)Be bulk metallic glasses isothermally annealed for time periods ranging from 10 to 600 min are shown in figures 1–3. The V1 samples were annealed at 613 and 633 K, i.e. below and above the caloric glass transition temperature T_g . The V4 samples were annealed at 643 K. All spectra of the

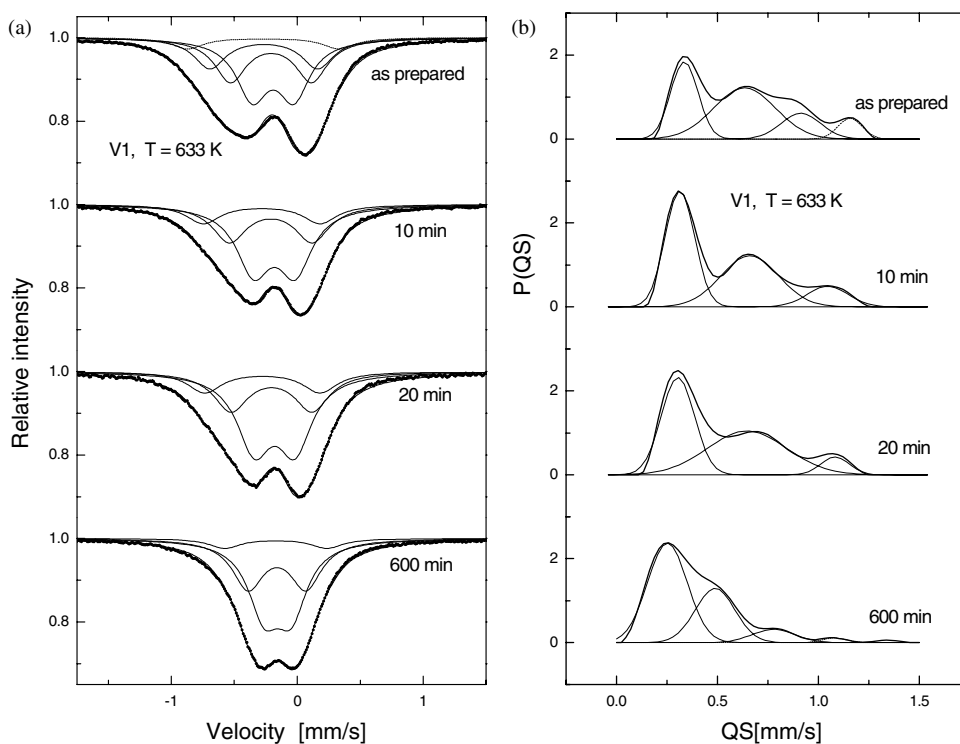


Figure 1. (a) Mössbauer spectra of V1 ($Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$) bulk metallic glasses annealed at 633 K (left) and (b) QS distributions (right).

samples consist of two broad asymmetric peaks characteristic for amorphous alloys. The data indicate the existence of an amorphous phase typically exhibiting quadrupole doublets with a distribution of the quadrupole splitting QS. The simplest form for the analysis of such spectra is the superposition of only a few quadrupole doublets, each originating from one of a few different types of Fe environment with different Mössbauer parameters. For the ZrTiCuNi(Fe)Be glasses, the minimum number of doublets that is needed to describe the experimental spectra by appropriate curve fitting is three. The results of the three- or four-doublet fits are shown in figures 1–3 as solid curves.

In order to overcome the problems of a fit with discrete single doublets, a distribution of hyperfine parameters was assumed for an improved fitting procedure. The Mössbauer spectra were analysed using the program of LeCaer and Dubois [10]. A linear dependence of the IS on the QS was assumed ($IS = a \times QS + b$). The quadrupole distributions $P(QS)$ derived from the best fitting curves to the measured spectra are also shown in figures 1–3. The data suggest that three (four) different types of environment around Fe atoms occur in the Zr-based amorphous alloys containing five different elements.

In the as-prepared states, the QS distributions show maxima at about 0.3, 0.6, 0.9 and 1.2 $mm\ s^{-1}$; lines mark the positions of the maxima. During the course of the isothermal annealing these peaks undergo characteristic changes. For the V1 glass the dominant peak at 0.3 $mm\ s^{-1}$ survives for short annealing times and is then shifted to 0.25 $mm\ s^{-1}$. The second peak at 0.6 $mm\ s^{-1}$ vanishes after 600 min of annealing and a shoulder at 0.5 $mm\ s^{-1}$ appears instead. The peak at 0.9 $mm\ s^{-1}$ disappears after short annealing times. The smallest peak at 1.2 $mm\ s^{-1}$ is still visible. The distributions corresponding to the highest annealing time,

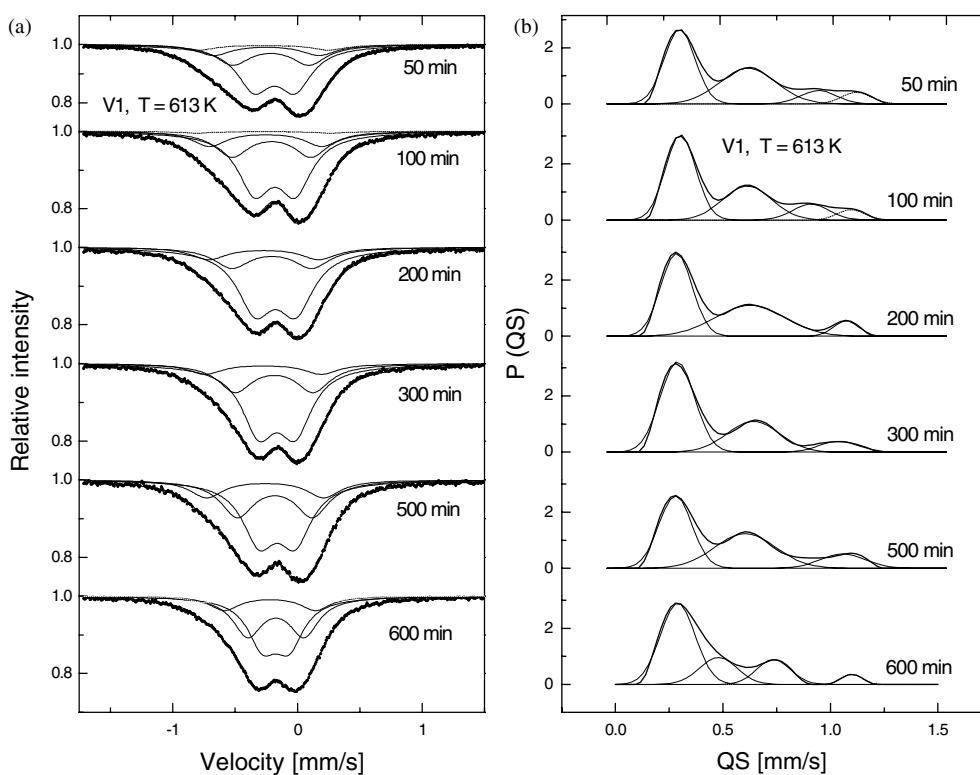


Figure 2. (a) Mössbauer spectra of V1 (Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni₁₀Be_{22.5}) bulk metallic glasses annealed at 613 K (left) and (b) QS distributions (right).

600 min, are different from the others. Within the $P(QS)$ curves for V4 glass no significant changes were observed during the annealing process. These results are in agreement with previous measurements for this alloy [11].

The asymmetry of the quadrupole doublet shows that there is a correlation between the IS and the QS. A linear relationship $IS = a \times QS + b$ was assumed where a and b were fitting parameters to minimize the sum of square deviations. The average values $\langle QS \rangle$ of the QS were taken as the centres of gravity of $P(QS)$ distributions. The corresponding $\langle IS \rangle$ values were calculated using $P(QS)$ values and the correlation mentioned above. Figure 4 shows the time dependence of the average $\langle QS \rangle$ and $\langle IS \rangle$ as a function of annealing time. The values of $\langle IS \rangle$ increase with annealing time while the $\langle QS \rangle$ decrease. This tendency is found mostly for the V1 glass. The spectra corresponding to the longest annealing time of 600 min are different to the others and therefore some changes in the hyperfine parameters of the Fe sites occur. An abrupt change of the IS (from -0.21 to -0.16 mm s⁻¹) and QS (from 0.60 to 0.36 mm s⁻¹) is observed for the sample annealed at the higher temperature of 633 K.

The same Mössbauer spectra were analysed using the RECOIL program of Rancourt and Ping [12]. In order to obtain a satisfactory fit, we had to use a sum of up to three Gaussian distributions with different widths. Here, a superposition with two sites has been used. One site has only one component (1 G) and the other site has two components (2 G), where all components of a site have the same IS relation $IS = a \times QS + b$. So this adds an additional degree of freedom as compared to the LeCaer method. The quality of the fits is slightly

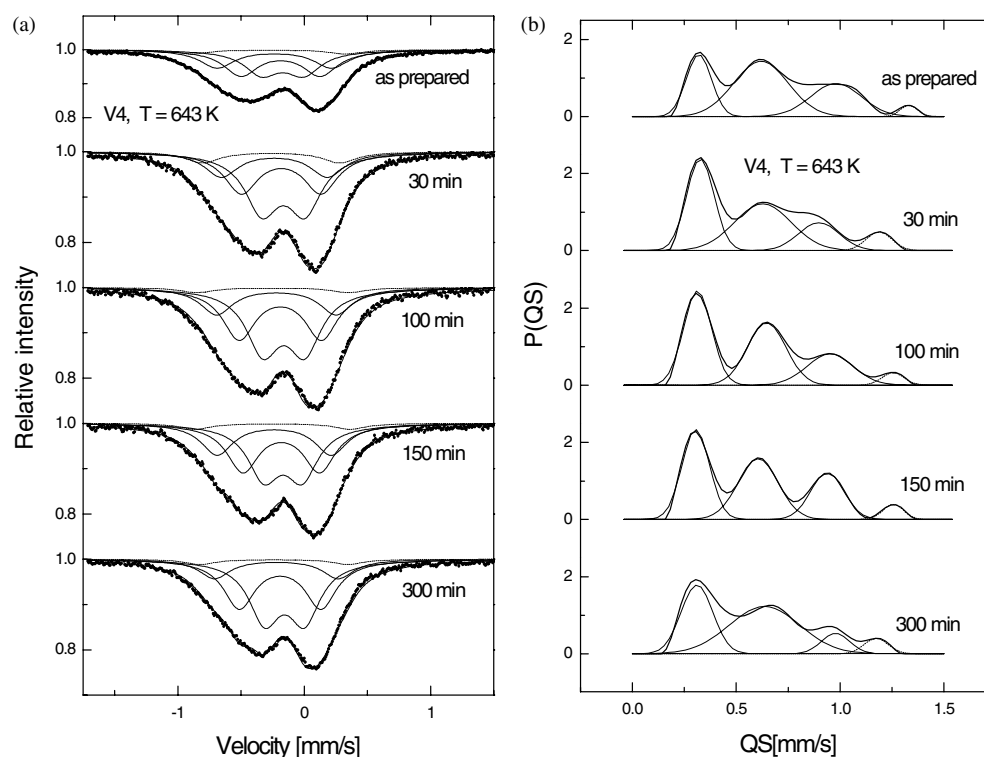


Figure 3. (a) Mössbauer spectra of V4 ($Zr_{46.7}Ti_{18.3}Cu_{7.5}Ni_{10}Be_{27.5}$) bulk metallic glasses annealed at 643 K (left) and (b) QS distributions (right).

improved. This result is close to the results obtained by Meyer *et al* [13] in ZrTiCuNi(Fe)Be where a sum of two Gaussian distributions was used and in contrast to ZrNiCuFe [14] where one distribution was sufficient to fit the asymmetric doublet.

The average values $\langle QS \rangle$ of the QS and mean centre shifts (IS) versus annealing time as obtained by the RECOIL program are shown in figure 5. The tendency of increasing the values of IS (IS) and decreasing the QS in V1 is repeated. The small difference in the (IS) values presented in figures 4 and 5 is caused by the two slightly different methods of fitting. The sum of Gaussian distributions with the same widths was used in the LeCaer method [10], while the widths of the Gaussian distributions in the RECOIL program of Rancourt and Ping [12] were variable and different. One site with two components (sites A and B) and the second site with one component (site C) always yield a satisfactory fit. All the results suggest that the Fe probes occupy three mean sites with different surroundings. Two of them (site A and B) with the smaller values of IS (around -0.17 mm s^{-1}) mainly contribute to the Mössbauer spectra. Site C has a more negative IS of around -0.5 mm s^{-1} and has only a small area fraction.

Figure 6 shows the time annealing dependence of the IS , QS and areas for these two dominant peaks of the $P(QS)$ distribution (sites A and B). The values of the IS for these two doublets increase with the annealing time, while the QS decrease. The biggest changes of the IS from -0.18 to -0.15 mm s^{-1} and QS from 0.34 to 0.26 mm s^{-1} for the first doublet and from $IS = -0.22$ to -0.16 mm s^{-1} and from $QS = 0.69$ to 0.51 mm s^{-1} for the second doublet are observed for the V1 glass annealed at 633 K. The main difference is observed in the areas of these two peaks in the $P(QS)$ distributions. During isothermal annealing the first

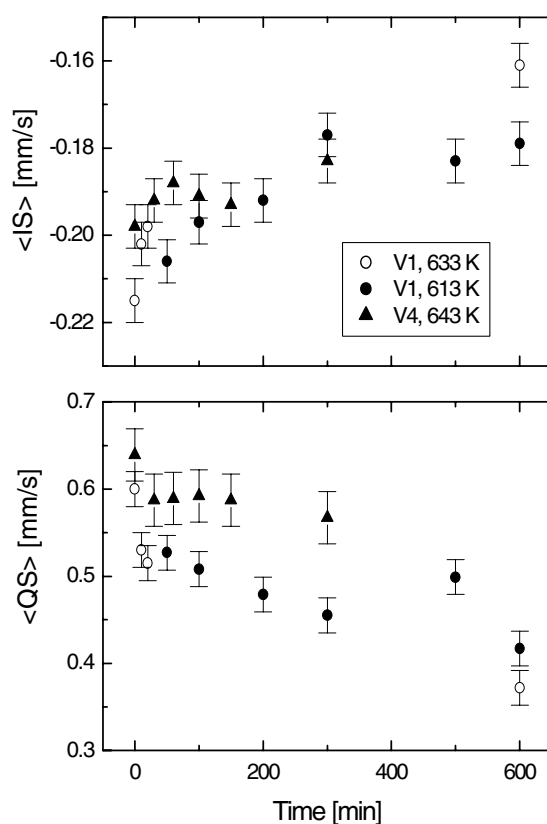


Figure 4. The average QS (QS) and IS (IS) as a function of the annealing time resulting from the Mössbauer analyses (LeCaer method).

peak at $QS = 0.34 \text{ mm s}^{-1}$ becomes dominant for V1. The percentage of this site A changes from 28% in the as-prepared sample to 63% in the sample annealed at 633 K for 600 min. The areas of these peaks for V4 glass do not change significantly. The third doublet (about 5%) with $IS = -0.50$ and $QS = 0.60 \text{ mm s}^{-1}$ which was added to obtain a satisfactory fit is independent of the alloy composition and annealing time. The tendency of an increasing area of the first peak (site A) at around $QS = 0.34 \text{ mm s}^{-1}$ in V1 was obtained by both the LeCaer methods and the RECOIL programme.

3.2. X-ray diffraction

A complementary XRD analysis of the Zr-based bulk amorphous alloys was performed. Figure 7 shows the wide-angle x-ray spectra of V1 annealed at 613 K for different times. After annealing for 300 min at 613 K this sample shows a slight sharpening of the second peak, this being probably related to a better adjustment of the short-range order in the amorphous phase. After 600 min annealing the slight sharpening of the second diffraction maximum becomes clearer and after 900 min the first diffraction peak also sharpens, indicating the formation of nanocrystals in the amorphous matrix. An XRD spectrum of the same sample after annealing at 633 K for 600 min is shown in figure 8. The shape of the peaks indicates the formation of a nanocrystalline phase in the glassy matrix. The alloy V4 was annealed at 643 K and the

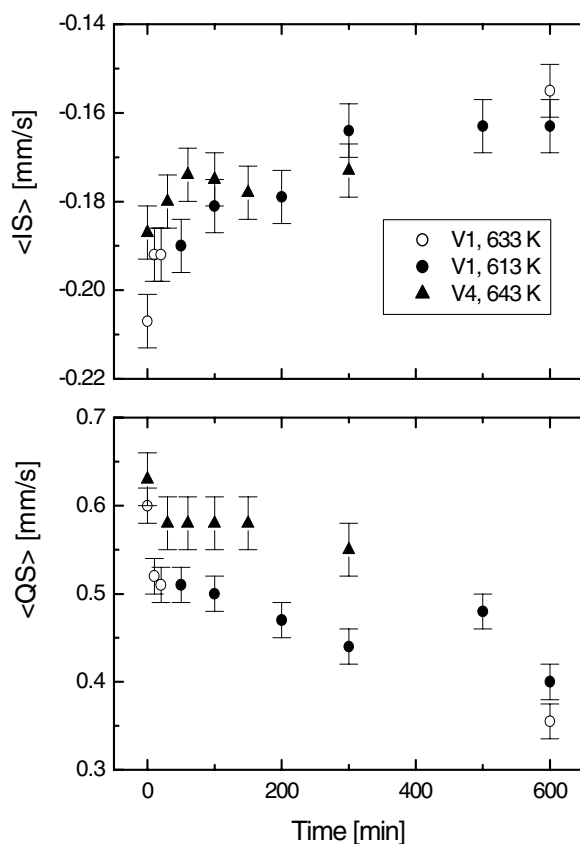


Figure 5. The average QS (QS) and IS (IS) as a function of the annealing time resulting from the Mössbauer analyses (RECOIL results).

XRD spectrum is presented in figure 9. After annealing for 300 min a sharpening of the first maximum occurs and as a sign of crystalline contributions sharp peaks occur in the annealed sample.

4. Discussion

Mössbauer spectra recorded during isothermal annealing demonstrate the presence of different local environments of Fe atoms, which undergo characteristic changes with increasing annealing time. The spectra and the $P(QS)$ distributions for the as-prepared samples of V1 and V4 are almost identical. This suggests that the Fe environments are similar in both alloys. The Zr-based bulk metallic glasses contain five different elements, and it is not possible to derive the arrangement of the atoms around a certain probe atom from the obtained hyperfine parameters.

In binary Zr–Fe amorphous alloys all IS values range between -0.3 and -0.1 mm s^{-1} and QS values between 0.25 and 0.60 mm s^{-1} [15–18]. A similar range for the QS has been reported for Fe-substituted Zr–Ni alloys [19, 20]. Another well known binary amorphous alloy—Ti–Fe—has IS of the same range as the Zr–Fe amorphous alloys with QS up to 0.40 mm s^{-1} [21]. Higher QS values of 0.65 mm s^{-1} have been observed by Michaelsen *et al* [18] in ^{57}Fe -

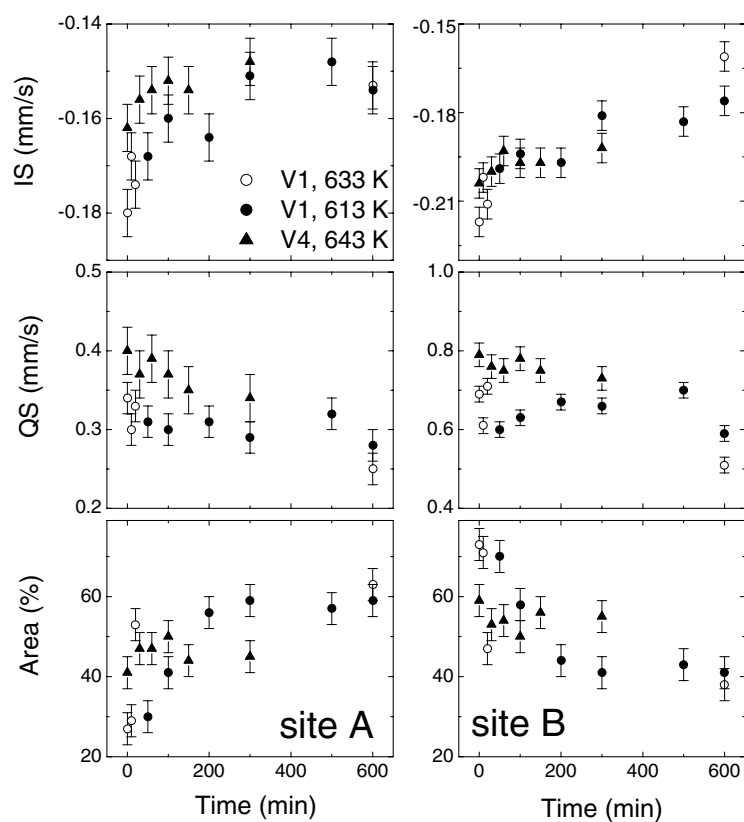


Figure 6. Time dependence of IS, QS and area fraction A for the two prominent quadrupole-split doublets of the V1 and V4 amorphous alloys (RECOIL results): left for site A and right for site B.

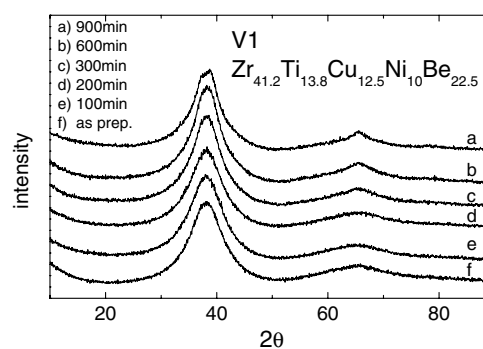


Figure 7. Wide angle x-ray spectra of V1 ($Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$) samples, annealed at 613 K for different times: (a) 900 min, (b) 600 min, (c) 300 min, (d) 200 min, (e) 100 min, (f) as prepared.

doped Zr–Cu amorphous alloys and by Mao *et al* [14] in the pseudo-binary metallic glass $Zr_{67}Ni_{10}Cu_{23}$ ($IS = -0.33 \text{ mm s}^{-1}$, $QS = 0.60 \text{ mm s}^{-1}$).

For the Mössbauer experiments, the absorbers of ZrTiCuNiBe alloys were prepared in which 1.5 at.% of Ni atoms were substituted by ^{57}Fe . We assume that Fe and Ni atoms have

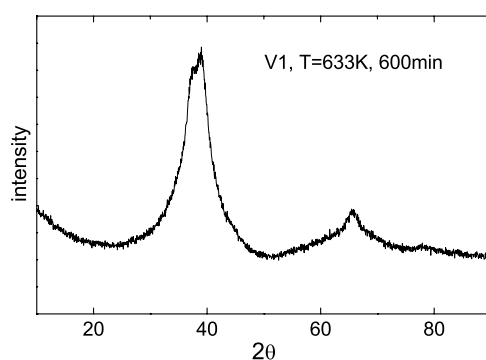


Figure 8. XRD spectrum of V1 ($\text{Zr}_{41.2}\text{Ti}_{13.8}\text{Cu}_{12.5}\text{Ni}_{10}\text{Be}_{22.5}$) samples, annealed at 633 K for 600 min.

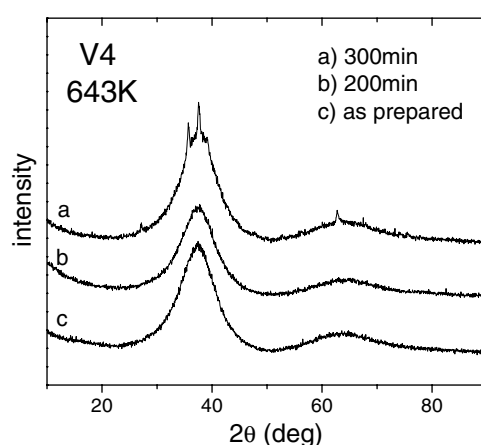


Figure 9. Wide-angle x-ray spectra of V4 ($\text{Zr}_{46.7}\text{Ti}_{18.3}\text{Cu}_{7.5}\text{Ni}_{10}\text{Be}_{27.5}$) samples, annealed at 643 K for different times: (a) 300 min, (b) 200 min, (c) as prepared.

similar chemical behaviour and also therefore do not significantly change the thermodynamical behaviour of the alloy. It seems to be reasonable to relate the peaks of the $P(QS)$ distribution to three different sites of Fe atoms with different surroundings in Zr-based bulk metallic glasses. The area ratio in the Gaussian distribution can be related to different stages of the decomposition process. The areas of the first and the second peak for V1 change during annealing; the first peak at $QS = 0.34\text{--}0.25 \text{ mm s}^{-1}$ becomes dominant. After 600 min of annealing at 633 K the contribution of the respective site A to the Mössbauer spectrum increases by a factor of two. SANS investigations reveal that Ti is involved in the decomposition process and that the decomposed regions are quasiperiodically arranged [8, 22]. These results match our hypothesis that the first dominant peak of the distribution $P(QS)$ is related mainly to Ti and Ni atoms surrounding the ^{57}Fe probe in a Ti-rich amorphous phase. The second peak with bigger QS 0.69 mm s^{-1} could be identified as an Fe site with Zr and Cu atoms in its local environment. We cannot exclude the existence of Be atoms around Fe atoms. ^{57}Fe atoms added to a Be sample cause in the Mössbauer spectrum a quadrupole doublet with $IS = -0.12 \text{ mm s}^{-1}$ and $QS = 0.56 \text{ mm s}^{-1}$ [23]. The similar peak observed in V1 might be related to Be-rich amorphous local environments. The third small additional doublet (5% area) has parameters close to Zr_2Fe , Zr_3Fe or Zr–Cu(Fe) amorphous alloys [18, 24–26]. The formation of Zr-rich

phases is a common feature in Zr-based alloys. The small changes of $\langle QS \rangle$ and $\langle IS \rangle$ in the course of isothermal annealing indicate small changes in the Fe environments, possibly as a result of ordering and rearrangements of the neighbourhoods.

We associate the abrupt change of the Mössbauer spectra for the V1 amorphous alloys when the values of $QS = 0.34$ and 0.69 mm s^{-1} change to 0.25 and 0.53 mm s^{-1} after annealing at 633 K for 600 min with a crystallization process. The XRD data show a slight sharpening of the first and second diffraction maxima after the formation of nanocrystals. At this time stage, cross section TEM clearly reveals the existence of nanocrystals embedded in an amorphous matrix [8]. During phase separation, composition and short-range order in at least one of the decomposed regions approach those of a competing crystalline phase and the nucleation probability increases rapidly. For the Zr–Ti–Cu–Ni–Be system, it has been shown that the crystallization temperature decreases with increasing Ti concentration [8]. Therefore we assume that the crystallization process begins in the Ti-rich amorphous phase.

XRD data of the V1 samples annealed at 613 K for more than 300 min only show a slight change in the short-range order, but after 900 min of annealing the sharpening of both diffraction maxima indicates the formation of nanocrystals in the amorphous matrix. In addition, SANS experiments have shown that these samples decompose into two amorphous phases after an incubation time of 150 min and form nanocrystals with a typical diameter of $2\text{--}3 \text{ nm}$ after annealing at 613 K for times longer than 600 min [22]. The IS and QS values are close to the values for the sample annealed at 633 K . After 900 min of annealing they come close to the other values (see figure 6). Possibly, these IS and QS values can be used to monitor the crystallization process.

The amorphous alloy V4 does not show decomposition before crystallization [9]. The ratio of the areas of the Gaussian distributions is found to be independent of the annealing time. The XRD pattern for the 300 min annealed sample shows crystalline contributions. Nevertheless, the Mössbauer spectra do not change, therefore Fe atoms are most probably not involved in the crystallization process.

5. Conclusions

Using ^{57}Fe atoms, the decomposition and crystallization processes in ZrTiCuNiBe alloys were studied by means of Mössbauer spectroscopy. The Gaussian distributions $P(QS)$ of individual spectra were resolved and analysed. The Mössbauer spectra of V1 ($\text{Zr}_{41.2}\text{Ti}_{13.8}\text{Cu}_{12.5}\text{Ni}_{10}\text{Be}_{22.5}$) and V4 ($\text{Zr}_{46.7}\text{Ti}_{8.3}\text{Cu}_{7.5}\text{Ni}_{10}\text{Be}_{27.5}$) alloys reveal the presence of three sites of Fe atoms with different local environments. The first peak of the Gaussian $P(QS)$ distribution is ascribed to a Ti-rich amorphous phase with Ti and Ni atoms around the Fe probe. The second peak might be related to a phase where the Fe atom is surrounded by Zr, Cu and Be atoms (Be-rich amorphous phase) and the third shows parameters close to Zr_2Fe , Zr_3Fe or $\text{ZrCu}(\text{Fe})$ alloys. The areas of the peaks only change in V1 during the decomposition process.

After annealing the V1 sample at 633 K for 600 min the first peak of the $P(QS)$ distribution becomes dominant. The values of IS and QS change drastically. The x-ray spectrum shows a slight sharpening of the diffraction maximum indicating the formation of nanocrystals embedded in the amorphous matrix. The crystallization process in the decomposed alloy begins in the decomposed Ti-rich amorphous phase. It is suggested that the nanocrystals mainly consist of Ti and Ni atoms. For the amorphous $\text{Zr}_{46.7}\text{Ti}_{8.3}\text{Cu}_{7.5}\text{Ni}_{10}\text{Be}_{27.5}$ (V4) alloy not showing decomposition, the hyperfine parameters do not change.

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