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# Free volume model: high temperature deformation of a Zr based bulk metallic glass

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## ABSTRACT

Homogeneous deformation of a zirconium based bulk metallic glass is investigated in the glass transition range. Compression tests at different temperatures and strain rates have been conducted. The mechanical behavior is analyzed in the framework of the free volume model, taking into account the dependence of the flow defect concentration with the deformation. The activation volume is evaluated and allows the gathering of the viscosity data (for the different strain-rates and temperatures) on a unique master curve. It is also shown that, due to the relation between flow defect concentration and free volume, it is not possible to deduce the equilibrium flow defect concentration directly from mechanical measurements. However, if this parameter is arbitrary chosen, mechanical measurements give access to the other parameters of the model, these parameters being of the same order of magnitude for the studied alloy when compared with other metallic glasses.

## KEYWORDS

Metallic glasses, high temperature deformation, plastic deformation, free volume model

## 1. Introduction

Interest in the mechanical behavior of metallic glasses, materials with no long-range order, has increased recently due to the development of alloys that can be obtained in amorphous state at low critical quenching rates. These new alloys are then able to be prepared as bulk specimens with size from mm to cm. The possibility to elaborate bulk specimens of metallic glasses (BMG), conversely to ribbons that were studied previously, means that reliable mechanical properties can now be investigated.

At high temperatures and/or low strain rates, metallic glasses deform in a homogeneous mode. In this mode, their mechanical-behavior has various characteristic features. The first one is the transition from a Newtonian flow (i.e. viscosity independent on strain-rate) to a non-Newtonian (i.e. decreasing viscosity when strain rate increases) behavior [1,2]. The second one is the possible existence of stress overshoots during uniaxial tests (compression and tension): typically, during a constant strain rate experiment, the measured stress reaches a maximum value before decreasing to a plateau value [3,4].

The question of the physical nature of the deformation mechanisms responsible for such behaviour is still a field of research. One of the most studied models is based on the free volume concept and on its mobility, as initially proposed for liquids by Cohen and Turnbull [5,6]. Free volume is defined as the volume in excess compared to an ideal disordered atomic configuration of maximum density. Spaepen extended to glasses the free volume model initially developed for liquids [7]. In this approach, the plastic deformation is due to uncorrelated atoms jumping in nearby local large enough holes or flow defects, namely with a size larger than a critical value. Such so-called flow defects have a concentration  $c_f$  given, according to the free volume theory, by:

$$c_f = \exp\left(-\frac{\gamma v^*}{v_f}\right) \quad (1)$$

where  $\gamma$  is a geometric factor that corrects the overlap between holes ( $0.5 \leq \gamma \leq 1$ ),  $v_f$  is the mean free volume, i.e. the average volume in excess per atom at a given temperature, and  $v^*$  is the critical size for which an atomic jump can occur. One can already note that the defect concentration is very sensitive to a slight variation in free volume concentration. Spaepen [7] suggested describing the plastic flow of metallic glasses in relation with the diffusion of atoms in the glass and the effect of stress on this diffusion. It leads to a relation between the flow stress and the plastic strain rate given by:

$$\dot{\epsilon} = 2c_f v_D \exp\left(-\frac{\Delta G^m}{kT}\right) \sinh\left(\frac{\sigma V}{2\sqrt{3}kT}\right) \quad (2)$$

where  $V$  is the activation volume for the stress bias. Uniaxial deformation implies the introduction of the Von Mises factor  $\sqrt{3}$  in the hyperbolic sinus. In this approach, the plastic strain rate results from the product of four terms: the Debye frequency ( $v_D$ ), the concentration of defect  $c_f$ , an Arrhenius equation including an activation free energy of defect migration ( $\Delta G^m$ ) and an hyperbolic sinus term which takes into account the effect of the applied stress on the energy barrier. From the relation between viscosity ( $\eta$ ), stress and strain-rate

( $\eta = \sigma/3\dot{\epsilon}$ ), the viscosity appears to be inversely proportional to the defect concentration. It was found that during an isothermal treatment at a given temperature  $T < T_g$ , the viscosity of the glass increases with time and finally reaches a plateau [8]. Considering that  $\eta \propto c_f^{-1}$ , such a behavior implies a decrease of the defect concentration before reaching an equilibrium value  $c_{f,eq}$ . This corresponds to structural relaxation. The change in defect concentration with respect to time is generally well described by a kinetic equation with a quadratic form [9]:

$$\dot{c}_f = -k_r c_f (c_f - c_{f,eq}) \quad (3)$$

where  $k_r$  is the rate constant of the structural relaxation, which is thermally activated. In a small temperature range, the variation of  $c_{f,eq}$  with temperature can be described by an Arrhenius law  $c_{f,eq} = c_0 \exp\left(-\frac{\Delta G^f}{kT}\right)$ , where  $\Delta G^f$  can be considered as the formation free energy of the flow defects.

In stress-strain curves of various BMG deformed in the homogeneous mode at high strain-rate and/or relatively low temperature, a softening behavior can be observed and understood by the creation of flow defects by plastic strain [3]. Such a view is also supported by experimental density measurements on a relaxed sample of a bulk Pd-based amorphous alloy, showing that plastic deformation can induce a significant density decrease of the glass [10].

De Hey et al. [3] have shown that it was possible to assume a linear dependence between the increments  $dc_f$  and  $d\epsilon$ , leading to a strain induced defect nucleation rate of the form:

$$P(c_f, \dot{\epsilon}, T) = a_x \dot{\epsilon} c_f \ln^2(c_f) \quad (4)$$

where  $a_x$  is a proportionality factor. From equation (3) and (4) the rate of variation of the defect concentration can be estimated as a balance between structural relaxation and strain-induced nucleation. This leads to the equation:

$$\dot{c}_f = -k_r c_f (c_f - c_{f,eq}) + a_x \dot{\epsilon} c_f \ln^2 c_f \quad (5)$$

In this framework, the steady state flow stress (i.e. stress plateau) corresponds to the stationary form of equation (5):  $\dot{c}_f = 0$ . The associated defect concentration  $c_f^*$ , in such a stationary regime, is given by the implicit form:

$$c_f^* = c_{f,eq} + \frac{a_x}{k_r} \dot{\epsilon} \ln^2 c_f^* \quad (6)$$

In equation (6), the stationary defect concentration is the sum of two terms. The first one ( $c_{f,eq}$ ) corresponds to the thermal equilibrium concentration of defects which depends only on temperature. This term can be qualified as a static component. The second term can be seen as a dynamic component since it corresponds to the increase in defect concentration resulting from plastic deformation. The link between free volume creation and annihilation, plastic deformation and thermodynamical properties of metallic glasses have been studied successfully in palladium [3], lanthanum [11] or zirconium [12] based metallic glasses.

In a recent study [13] has been introduced the possibility to model entirely compression stress-strain curve obtained at high temperature. However, the determination of different parameters was not rigorous. The aim of this study is to investigate deformation behavior of a ZrCuAlTiNi BMG for temperatures inside and slightly above the glass transition range (in the super liquid region), to discuss the effects of temperature and strain-rate on viscosity or flow stress in the framework of the free volume model and to propose a method to determine different parameters of the free volume model thanks to compression measurements only based on a study of the steady-state as described by the free-volume theory.

## 2. Experimental procedure

A glass of composition  $Zr_{52.5}Al_{10}Cu_{22}Ti_{2.5}Ni_{13}$  was elaborated under the form of 6 mm rods. The pure metals were melted in a cold crucible in an argon atmosphere and injected under pressure in a water-cooled copper mould. The degree of amorphisation was characterized by X-ray diffraction (copper wavelength, using  $\theta$ - $2\theta$  goniometer or four circles apparatus). Differential Scanning Calorimetry (DSC) scans with a heating rate of 10 K/min under argon atmosphere were performed to study the thermal stability of the glass. Cylindrical samples of 5 mm diameter and 8 mm height were used for the compression tests. These tests were carried out between 683 K and 703 K, with a heating rate of 10 K/min up to the test temperature and for strain-rates ranging from  $2.5 \times 10^{-4} \text{ s}^{-1}$  to  $5 \times 10^{-3} \text{ s}^{-1}$ . Constant strain-rate (mainly  $5 \times 10^{-4} \text{ s}^{-1}$ ) and strain-rate jumps tests were performed.

## 3. Results

### 3.1 Studied glass

X-ray spectrum of the as-prepared glass has already been published in [13]. It confirms that the alloy is obtained under the amorphous state. The characteristic temperatures (glass transition temperature  $T_g$  and crystallization temperature  $T_x$ ) observed during a DSC thermal scan are shown in Figure 1:  $T_{g1} = 659 \text{ K}$  ( $T_{g1}$  is the onset of the glass transition) and  $T_x = 761 \text{ K}$ ,  $\Delta T = T_x - T_g = 102 \text{ K}$ . These values ( $T_x$ ,  $T_g$  and  $\Delta T$ ) can be compared to those encountered in the case of Vitrelloy 4. This glass, known for his excellent aptitude to amorphisation, possesses a  $T_g = 622 \text{ K}$  and  $T_x = 727$ , thus  $\Delta T = 105 \text{ K}$  [14]. All these temperatures are of the same order of magnitude as the ones measured for the present glass.

### 3.2 Mechanical tests

Compression tests have been conducted between 683 and 703 K, i.e. in the glass transition range, according to DSC results performed with the same heating rate. The strain rates used allow, in that temperature range and for this glass, to study the transition from a Newtonian to a non-Newtonian deformation mode.

Typical results of compression experiments at a strain rate of  $5 \times 10^{-4} \text{ s}^{-1}$  are presented in figure 2 for three different temperatures. The flow stress is strongly thermally activated: it decreases from almost 225 MPa to 25 MPa when the temperature increases of 20 K only. Large plastic deformation ( $\epsilon \approx 1.0$ ) can be reached without extensive hardening (even if a slight hardening can be detected), which would result from crystallization. This was

confirmed by X-ray measurements and transmission electron microscopy observations performed after deformation showing no trace of crystallization.

Figure 3 presents typical results of strain-rate jump tests obtained at 683 K with successively increasing and decreasing step. These experiments display several interesting features. Firstly, one can note the high strain rate sensitivity of the flow stress. Secondly, the steady state flow stress depends only on the applied strain-rate, but not on the past mechanical history of the sample. Finally, for the highest strain-rates, stress overshoots or undershoots are detected after the strain rate increase or decrease respectively.

## 4. Modeling and discussion

### 4.1 Constant defect concentration at a given temperature: determination of activation volume

In this section are presented the calculation of the activation volume  $V$  assuming a low variation of the flow defect concentration. Under this assumption – to be validated hereafter, defect concentration depends only on temperature. Since experiments were carried out in the range of the glass transition, the defect concentration when deformation starts is supposed to be equal to its equilibrium value (i.e.  $c_f^* = c_{f,eq}$ ). In such a framework, equation (2) can be rewritten:

$$\dot{\epsilon}_T = \dot{\epsilon}_0 \sinh\left(\frac{\sigma V}{2\sqrt{3}kT}\right) \quad (7)$$

where  $\dot{\epsilon}_0(T) = 2c_{f,eq}v_D \exp\left(-\frac{\Delta G^m}{kT}\right)$  is a constant at a given temperature. The activation volume is the volume of matter involved in each elementary defect jump. The simplest way to evaluate  $V$  is to fit the sinh law in the purely plastic state. When overshoots are detected, the value of the flow stress used is its maximum value preceding the overshoot, which corresponds to a stationary plastic strain-rate with the initial flow defect concentration, since at this maximum  $\dot{\sigma}/E = 0$ , and not the stress plateau level following the overshoot where the flow defect concentration has relaxed.

As can be seen in figure 4 (plain curve and circles), good fits of equation (7) with experimental results can be obtained with the values of  $V$  and  $\dot{\epsilon}_0$  given in table I. Activation volumes between 193 and 206 Å<sup>3</sup> are measured, depending on the test temperature. Such values are in agreement with previously published data dealing with high temperature deformation of various metallic glasses [3,15] but are higher than directly expected from the free volume model. As a matter of fact, this model postulates that atoms jump from their site into a nearby vacancy-like hole, which implies an activation volume of about one atomic volume. Using the mean atomic volume of the studied glass ( $\Omega \approx 13 \text{ Å}^3$ ), such a measured activation volume corresponds instead to the displacement of 10 to 20 atoms. This result supports the idea that atoms do not jump without correlated displacements of their neighbors, phenomenon also observed in diffusion experiment, see [16] for a review. Then, the measured activation volume corresponds to the volume of matter in which atoms are displaced during the elementary shear event, multiplied by the corresponding mean atomic strain. If we consider the existence of a spherical strain gradient of mean value  $\bar{\epsilon}$  around a jumping atom

implying  $N$  atoms, the work done by the applied stress is  $W = \sigma V = \sigma \Omega N \bar{\epsilon}$  and the effective activation volume is:  $V = N \Omega \bar{\epsilon}$ . Taking, for instance,  $\bar{\epsilon} \approx 0.5$  leads to  $N \approx 30$  atoms would be implied in an elementary plastic event.

The measured values of  $\dot{\epsilon}_0$  vary from  $2.6 \times 10^{-4} \text{ s}^{-1}$  to  $3.4 \times 10^{-3} \text{ s}^{-1}$ . This parameter includes several contributions: the equilibrium defect concentration  $c_{f,eq}$ , the free energy of atomic migration and the Debye frequency. It can be rewritten extracting explicitly enthalpy and entropic terms, according to:

$$\dot{\epsilon}_0 = \left[ 2\nu_D c_0 \exp\left(\frac{\Delta S^m + \Delta S^f}{k}\right) \right] \exp\left(-\frac{\Delta H^m + \Delta H^f}{kT}\right) \quad (8)$$

where  $\Delta S^f$  and  $\Delta S^m$  are respectively the formation and migration entropies of the free volume defects, and  $\Delta H^f$  and  $\Delta H^m$  their formation and migration enthalpies. With this formalism, it is found  $\Delta H \approx 5.4 \text{ eV}$  and  $\left[ 2\nu_D c_0 \exp\left(\frac{\Delta S}{k}\right) \right] \approx 4 \times 10^{36} \text{ s}^{-1}$ . In the case of a  $Zr_{55}Cu_{30}Al_{10}Ni_5$  glass, a value  $\Delta H \approx 4.2 \text{ eV}$  was obtained [12]. Concerning the frequency factor, such high values have been already reported for BMG. In the case of a Pd-Ni-P glass, values between  $10^{34}$  and  $10^{42} \text{ s}^{-1}$  were reported [17]. Such high values can be interpreted as the signature of a cooperative phenomenon, through a high value of the entropic parameter [16,17]. This short analysis supports the assumption that flow defects in metallic glasses imply ten or more moving atoms.

From equation (7), the viscosity can be written as:

$$\eta = \frac{\sigma}{3\dot{\epsilon}} = \frac{\sigma}{3\dot{\epsilon}_0 \sinh\left(\frac{\sigma V}{2\sqrt{3}kT}\right)} \quad (9)$$

At low stress, equation (9) leads to the Newtonian viscosity  $\eta_N$ :

$$\eta_N = \frac{2\sqrt{3}kT}{3\dot{\epsilon}_0 V} \quad (10)$$

From a combination of equations (9) and (10), a master curve can be derived to gather the variations with strain rate of the viscosity at different temperatures. Indeed, if  $\dot{\epsilon}_0$  depends only on temperature (implying that  $c_f$  does not vary with strain and strain-rate, as assumed in this paragraph),  $\eta/\eta_N$  is given by:

$$\frac{\eta}{\eta_N} = \frac{\sigma V / 2\sqrt{3}kT}{\sinh(\sigma V / 2\sqrt{3}kT)} = \frac{x}{\sinh(x)} \quad (11)$$

with  $x = \sigma V / 2\sqrt{3}kT$ . Figure 5 (already published in [13]) displays the variation of  $\eta/\eta_N$  with  $x$ . All experimental data performed at different temperatures and strain-rates superimpose with a good precision on a unique master curve. This agreement confirms the assumption made previously of a constant defect concentration for the activation volume measurements.

#### 4.2 Defect concentration as a function of strain-rate: determination of kinetic parameters

As previously mentioned, in steady state conditions, the flow defect concentration is expected to vary with strain-rate. If a dynamical equilibrium defect concentration is established, a constant steady state flow stress is obtained. These steady state conditions are associated to  $\dot{c}_f = 0$ . Then the stress strain-rate relation is described by the set of equations:

$$\dot{\epsilon} = c_f^* \dot{\epsilon}_{0,c} \sinh\left(\frac{\sigma V}{2\sqrt{3}kT}\right) \quad (12)$$

$$c_f^* = c_{f,eq} + \frac{a_x}{k_r} \dot{\epsilon} \ln^2(c_f^*) \quad (13)$$

$$\text{with } \dot{\epsilon}_{0,c} = v_D \exp\left(-\frac{\Delta G^f}{kT}\right).$$

One can note from equations (12) and (13) that the non-Newtonian behavior of metallic glasses, which is promoted by a decrease of temperature or an increase of strain-rate, can have two origins. A first deviation from a Newtonian flow results from the direct effect of the stress on the atomic energy landscape, and a second deviation may be due to the free volume increase by plastic deformation.

Thanks to equations (12) and (13) and to the experimental stress/strain-rate plateau, it is possible to estimate the parameters  $a_x/k_r$  and  $\dot{\epsilon}_{0,c}$  if  $c_{f,eq}$  is known:

$$\frac{a_x}{k_r} = \frac{\dot{\epsilon}_{0,c} \sinh\left(\frac{\dot{\epsilon}}{\dot{\epsilon}_{0,c} \sinh\left(\frac{\sigma V}{2\sqrt{3}kT}\right)}\right)^{-c_{f,eq}}}{\dot{\epsilon} \ln^2\left(\frac{\dot{\epsilon}}{\dot{\epsilon}_{0,c} \sinh\left(\frac{\sigma V}{2\sqrt{3}kT}\right)}\right)} \quad (14)$$

By evaluating these parameters and including the steady state flow defect concentration, it becomes possible to fit the plateau values (strain-rate vs. stress) of the compression experiment, as illustrated by figure 4 (dotted curve and squares) with a good agreement. The difference in stress on figure 4, between plain and dotted curve, illustrates the effect of the taking into account of flow defect concentration variation as an increase of non-Newtonian nature of the glass rheology. In practice, one can also note that the determination of the parameters  $a_x/k_r$  and  $\dot{\epsilon}_{0,c}$  necessitates that experimental stress-strain curve present at least one important overshoot (i.e. a decrease of the stress of approximately 20 to 30 % at least) in order to obtain physically coherent values. In the high temperature case where there was no trace of overshoot, it was impossible to determine a stable value of these parameters, which could even take non-physical negative values.

However, due to the non-linearity of equation (14), various  $(a_x/k_r, c_{f,eq}, \dot{\epsilon}_{0,c})$  triplets allow a good fit of the experimental data. For the sake of illustration, typical results at 683 K are given in table (3) with values of  $c_{f,eq}$  differing from more than five range orders (between  $9.1 \times 10^{-16}$  and  $6.2 \times 10^{-10}$ ). This means that it is practically difficult to identify the parameters  $(a_x/k_r, c_{f,eq}, \dot{\epsilon}_{0,c})$  directly from mechanical testing. Nevertheless, all the predicted triplets lead almost exactly to the same relative variation of the defect concentration for a given change in strain-rate at a given temperature, as illustrated by figure 6. This means that the ratio of the



static over the dynamic contributions in equation (6) remains also constant, whatever the triplet. In other words, the choice of  $c_{f,eq}$  determines directly and without ambiguity the values of the parameters  $a_x/k_r$  and  $\dot{\epsilon}_{0,c}$ . Figure 7 presents the dependency of these two parameters with  $c_{f,eq}$  deduced from experimental results at 693 K. As one can see in figure 7, the ratio  $a_x/k_r$  is roughly proportional to  $c_{f,eq}$  whereas  $\dot{\epsilon}_{0,c}$  is inversely proportional to  $c_{f,eq}$ . This indicates that mechanical testing cannot lead to a direct measure of the defect concentration, but rather to the determination of its relative variations.

A similar conclusion about the uncertainty on the measurement of  $c_{f,eq}$  was also drawn from a study of the thermal relaxation behavior of a  $Zr_{55}Cu_{30}Al_{10}Ni_5$  BMG [12]. In this work, Daniel et al. performed two series of DSC experiments on samples pre-annealed for various times and temperatures. On the basis of these experiments, an optimization procedure was carried out to identify a set of parameters allowing the simulation of the experimental variations with temperature of the apparent specific heat. Several sets of parameters for the variation with temperature of the defect concentration equally fitted the DSC measurements. It was concluded that the free volume model was able to successfully describe the thermal relaxation behavior of the glass but with a possibility of variation of at least five range orders for  $c_{f,eq}$  at a given temperature. In other words, the uncertainty concerning  $c_{f,eq}$  could not be overcome by DSC measurements. This practical difficulty is directly related to the exponential law linking the free volume and the defect concentration (equation (1)). For instance, the values of  $c_{f,eq}$  used in table II (about six range of orders between the minimum and maximum values) corresponds only to a variation of about 50 % on the value of the free volume concentration (from about 0.029 to 0.047).

Finally, if one takes into account published values of  $k_r$  for a  $ZrCuAlNi$  BMG heated at similar  $T/T_g$  ratio [12] as in the present study, the values of  $a_x/k_r$  and  $\dot{\epsilon}_{0,c}$  lead to values of  $a_x$  between  $10^{-3}$  and few  $10^{-2}$ . Such values are in relative agreement with previously reported ones [11,18]. Moreover, in this study, the predicted variation of the flow defect concentration ( $c_f^*/c_{f,eq}$ ) is approximately 2.6 at  $\dot{\epsilon} = 10^{-2} \text{ .s}^{-1}$ , which is also consistent with published results [3,11].

## 5. Conclusions

The homogeneous deformation in the supercooled domain of a  $ZrCuAlNiTi$  BMG has been studied in the framework of the free volume model. High values of the activation volume of the plastic deformation indicate a cooperative motion of a group of a few tens of atoms per elementary plastic event. The validity of the activation volume analysis and measurement ensures the validity of a scaling law of the glass viscosity versus strain-rate and temperature.

From the mechanical tests, it is possible to estimate the relative variation of the steady state defect concentration with strain-rate and, knowing the equilibrium defect concentration and the activation volume, a method was proposed to determine  $a_x/k_r$  (the ratio of the strain induced defect creation rate over the recovery constant) and  $\dot{\epsilon}_{0,c}$  (migration rate). Because of the exponential law which links free volume and flow defect concentration, it is impossible to determine the absolute value of the thermal equilibrium defect concentration  $c_{f,eq}$  itself. The great number of solutions of the mechanical constitutive equations of the glass leads however to the same ratio of the static (thermal) part over the dynamic (strain and recovery affected) part of this defect concentration, in agreement with other BMG data.

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## Figures captions

- Figure 1 Typical DSC curve for D' glass, with  $\dot{T}=10$  K/s.
- Figure 2 True stress vs. true strain curves for three different temperatures (683, 693 and 703 K) at constant strain-rate ( $5 \times 10^{-4} \text{ s}^{-1}$ ).
- Figure 3 True stress vs. true strain curve at 683 K. The strain-rate jump measurement are:  $2.5 \times 10^{-4}$ ,  $5 \times 10^{-4}$ ,  $10^{-3}$  and  $2.5 \times 10^{-3} \text{ s}^{-1}$ , and then goes back to  $5 \times 10^{-4} \text{ s}^{-1}$ .
- Figure 4 Strain-rate vs. stress at 683 K. Circles: experimental values of the maximum stresses, plain line: fit of equation (8). Squares: experimental values of the steady state, dotted line: fit obtained with set of equations (12) and (13).
- Figure 5 (a) Viscosity vs.  $\dot{\epsilon}$  for three temperatures.  
(b) Deduced master curve for the normalized viscosity, according to equation (12) (already published in [13]).
- Figure 6 Relative variation of the defect concentration for the three set of parameters presented in table 2 as a function of strain rate according to equation (6).
- Figure 7 Variation of the ratio  $a_x/k_r$  and  $\dot{\epsilon}_{0,c}$  with  $c_{f,eq}$ .

## Tables

T (K)	V ( $\text{\AA}^3$ )	$\dot{\epsilon}_0$ ( $\text{s}^{-1}$ )
683	201	$2.6 \times 10^{-4}$
693	206	$9.2 \times 10^{-4}$
703	193	$3.4 \times 10^{-3}$

Table I.

Activation volume and frequency factor's values deduced from experimental results.

$x_{\text{eq}}$	$c_{f,\text{eq}}$	$a_x/k_r$	$\dot{\epsilon}_{0,c}$
0.0289	$9.07 \times 10^{-16}$	$1.29 \times 10^{-16}$	$2.7 \times 10^{11}$
0.0328	$5.55 \times 10^{-14}$	$1.02 \times 10^{-14}$	$4.4 \times 10^9$
0.0472	$6.20 \times 10^{-10}$	$2.39 \times 10^{-10}$	$3.4 \times 10^5$

Table II.

Example of three sets of parameters fitting well the experimental data at 683 K,  $x_{\text{eq}}$  and  $c_{f,\text{eq}}$  have been inserted for the sake of comparison of their relative variation.

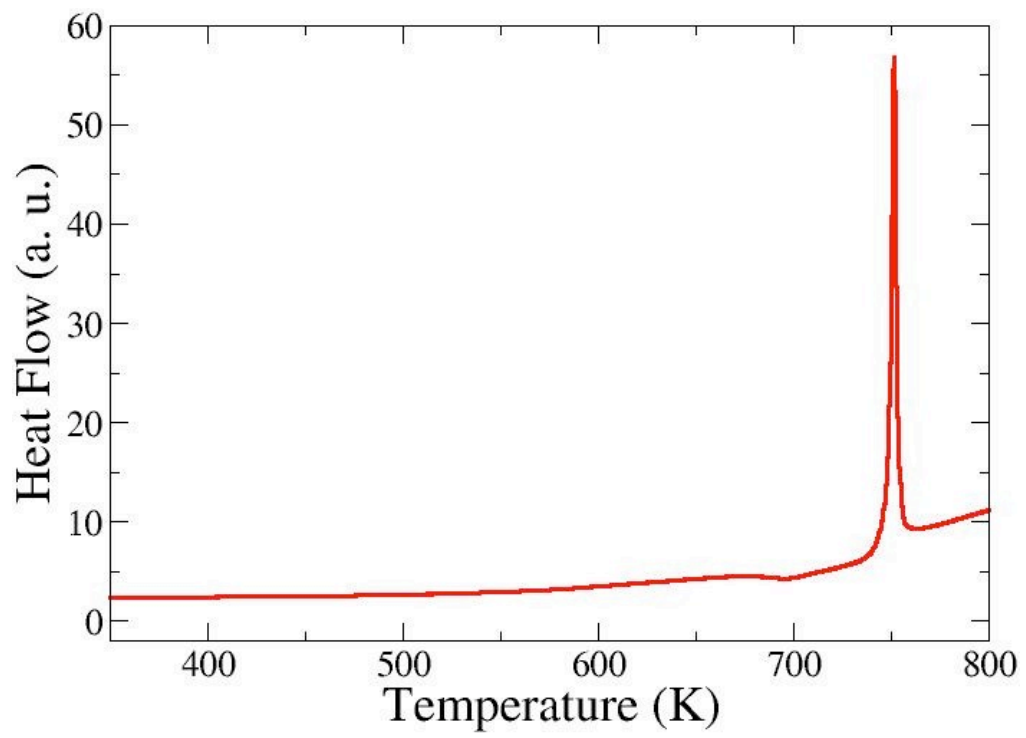


Figure 1

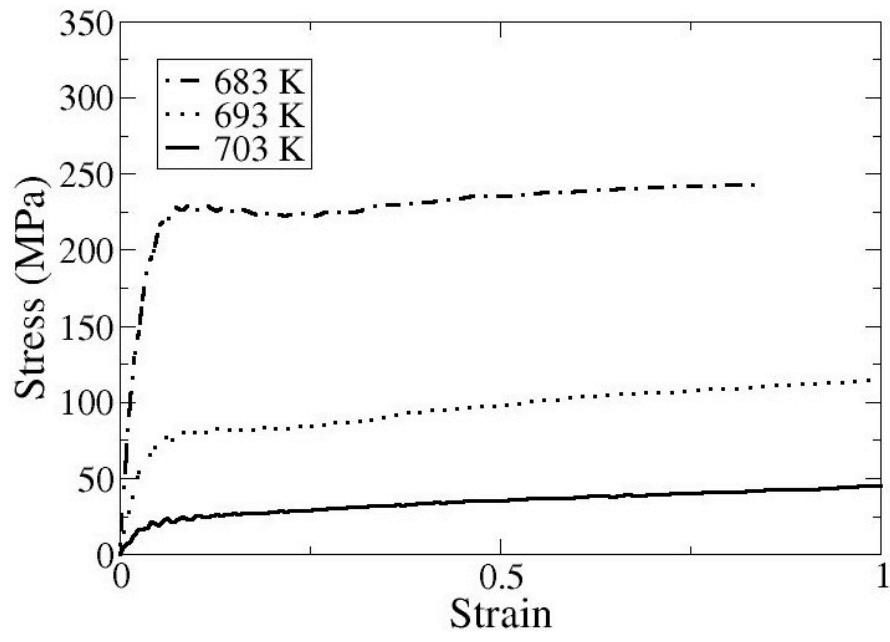


Figure 2

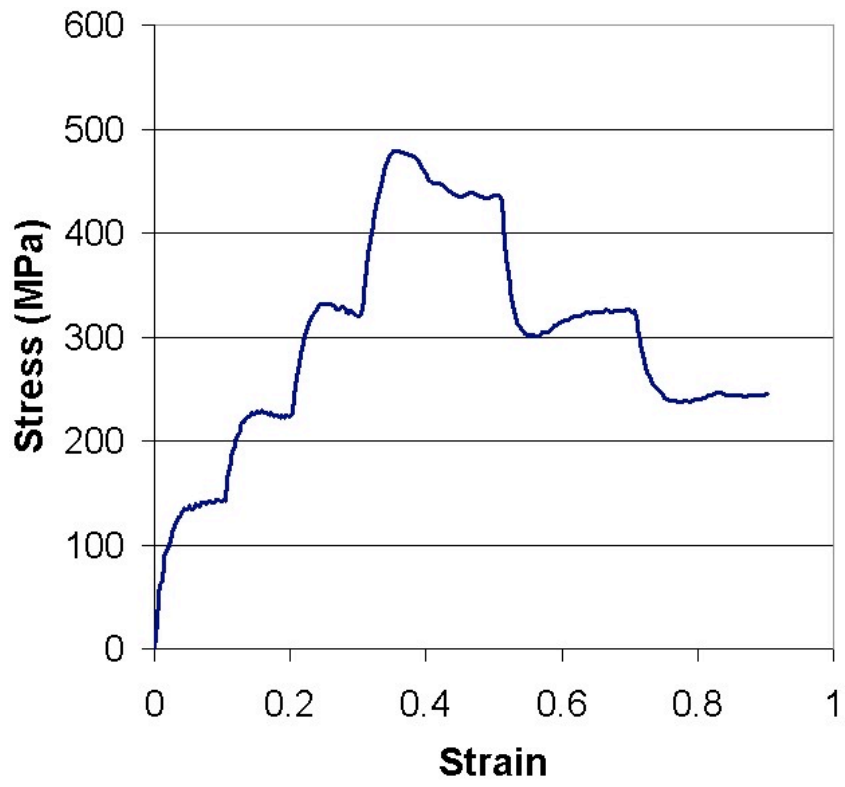


Figure 3

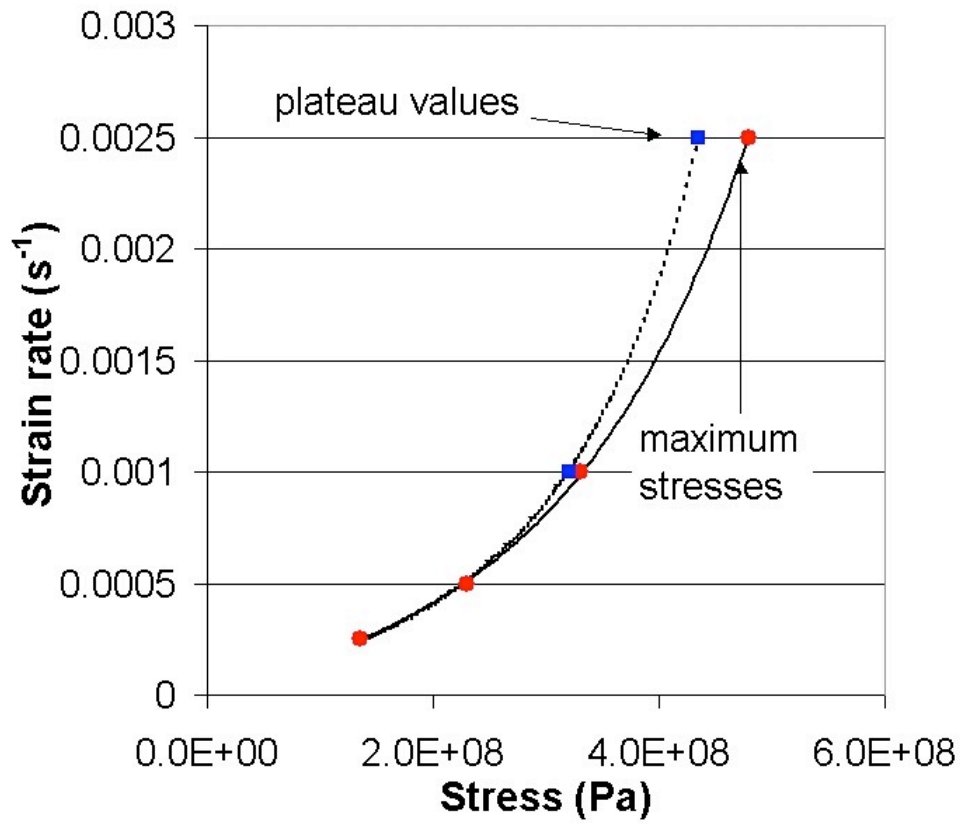


Figure 4



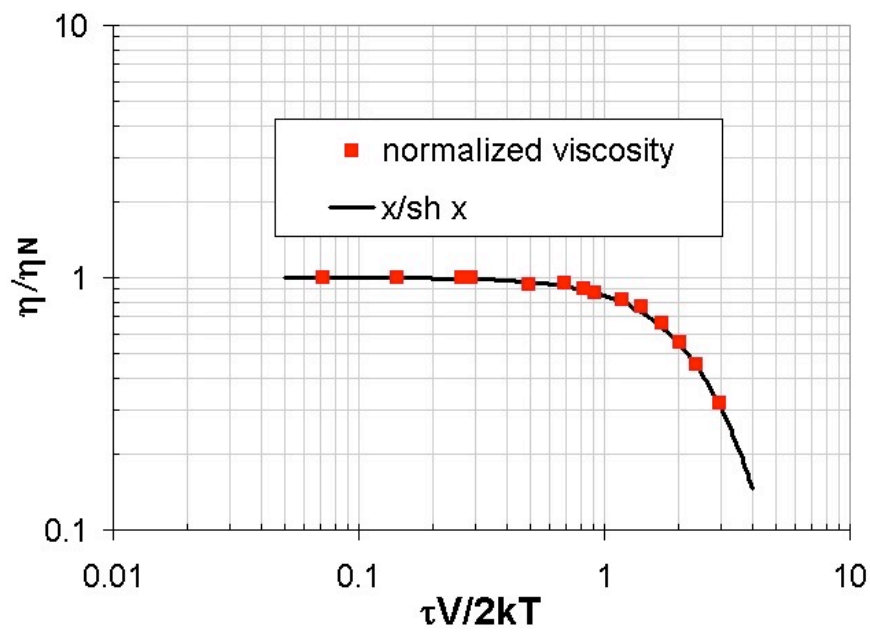
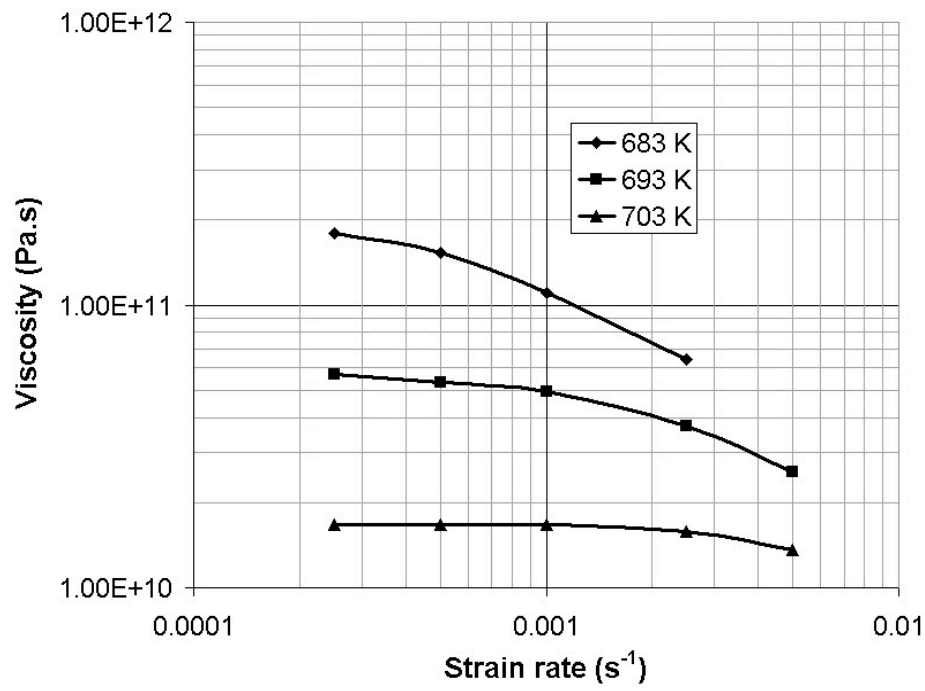


Figure 5 a and b

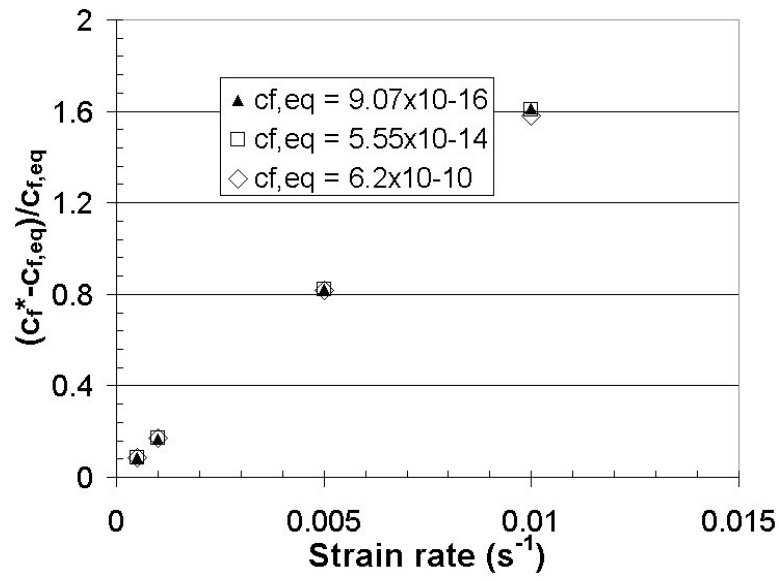


Figure 6

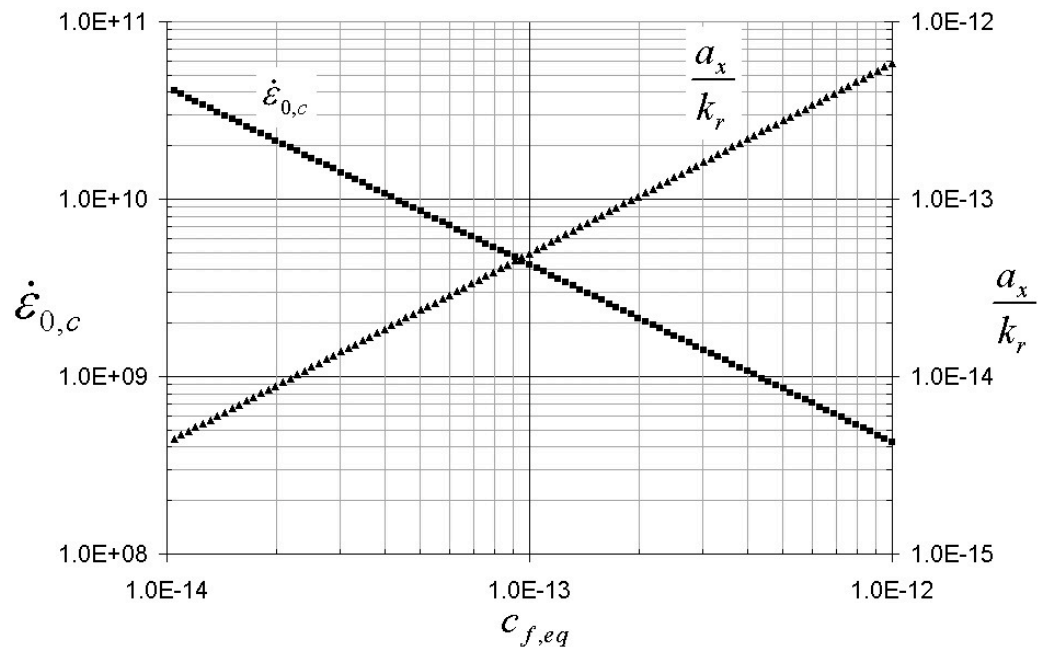


Figure 7