

NORMALISING THE VALUES OF ISOTHERMAL DILATOMETRIC
MEASUREMENTS OF AMORPHOUS METALLIC SAMPLES

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Abstract

Dilatometric experimental technique /1/ represents specific method with which it is possible to observe the kinetics of low-temperature relaxation of amorphous metallic ribbons. The technique requires exceptional accuracy so that the contraction of samples under the influence of rearrangement of atoms which changes the excess of free volume can be observed.

The development of this technique required a series of additional corrections to increase the precision of measurements and to achieve a realistic picture of experimental results. This paper describes the improvement of this experimental technique in terms of comparisons of the results obtained. In the paper we present the measurement of the length of amorphous system $\text{Co}_{78}\text{Nb}_{16}\text{B}_6$ using the new improved experimental technique.

Introduction

It is known that experimental results of isothermal dilatometric investigation of amorphous samples are obtained so that the sample is brought to isothermal temperature in a short time and than allowed to relax. The process of sample transformation at a given temperature is accompanied by its thermal expansion which is one or two orders of magnitude larger than contraction of sample which occurs later under the influence of annealing the excess free volume. The method is very well known and it is given in /1/.

The contraction of sample, at least at the very beginning, in most cases develops according to the logarithmic dependence /2/.

If we observe the isothermal change of length in the presence of relatively low stresses, at the beginning the process of isothermal contraction is going to be dominant. The process

of contraction will thus not be influenced by the isoconfigurational flow, because /among the other things/ it is period of cluster formation.

The results obtained in a well arranged experiment follow very well the logarithmic dependence of time change on the isothermal process duration. But, it is very difficult to define the relationship between two different measurements. Actually, we know only how much each sample contracted, but when we investigate two samples it is very difficult to define which contraction is greater, in other words it is difficult to compare the free volumes of the samples investigated. That is the reason why we can't define the initial conditions which describe the kinetics of the low-temperature relaxation process.

The situation is rather complex since the functional dependence is of logarithmic nature, and that means that the function is indefinite for the initial moment $t=0$.

Experimental measurement and reduction to the same initial conditions

During the investigation of the amorphous system $Co_{78}Nb_{16}B_6$ with the help of the isothermal technique the results obtained are shown in figure 1. These results were obtained from measurements on samples which were exposed to different mechanical relaxation. The sample (1) was subjected to a stress of 30 MPa.

Sample	a	b	Coeff.Regr.
1.	33.53045	-4.56973	0.99553
2.	39.37242	-5.81024	0.99509
3.	44.51188	-6.50964	0.99884

sample (2) with a stress of 50 MPa and sample (3) with 60 MPa. The lengths of samples were 17 mm and isothermal temperature was $210^{\circ}C$. The graphs were obtained using the standard methods, i.e. measurements are made from the moment the isothermal contraction begins.

It is obvious that all three graphs show distinct logarithmic dependence but they don't tell us about relationships between them. By fitting the first ten measurements to the logarithmic expression $y = a + b \ln t$ the results shown on the table

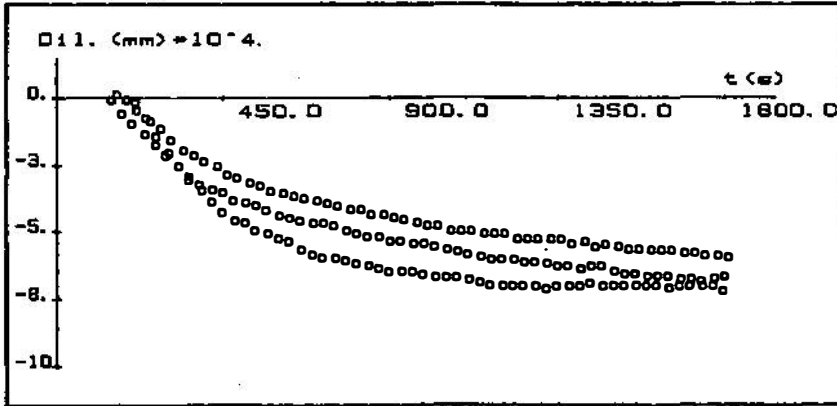


Figure 1.

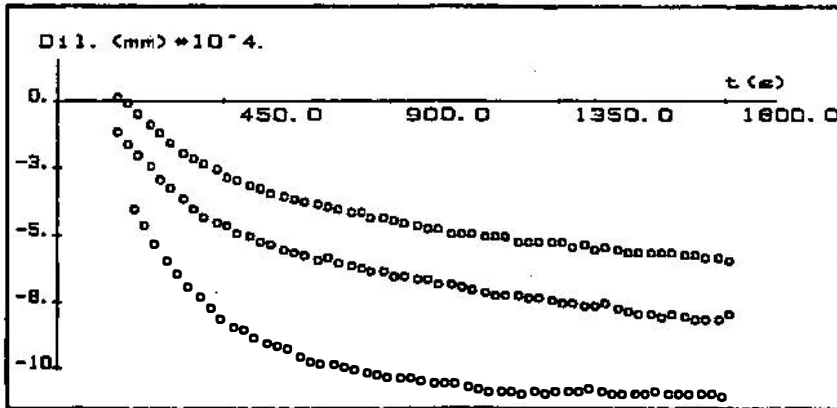


Figure 2.

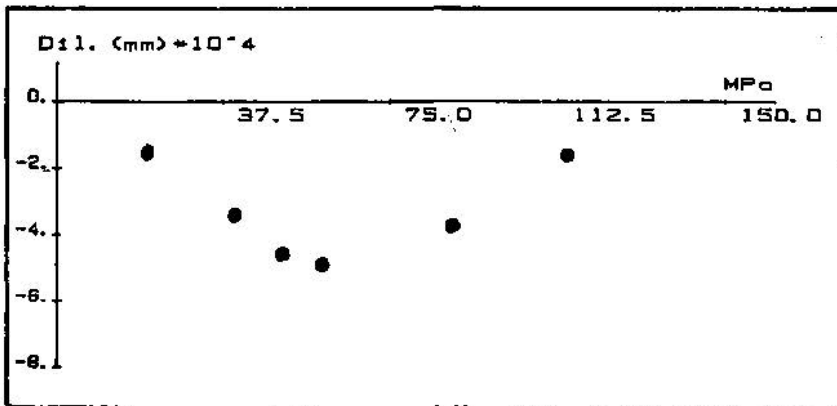


Figure 3.

were obtained. An exceptionally good logarithmic agreement is observed.

To reduce the measurements on different samples in some way to the mutually comparable results we came upon the idea to draw secants and then to correct the results so that the intersection for all the samples with the ordinate axis would be the same. This was done and the results are shown on figure 2. The advantage of the correction procedure can be seen when fig. 1 and 2. are compared. It is not difficult to notice that from fig.1 we cannot see the correlations of the values which the individual measurements give. From fig.2, however, it can be clearly seen which sample contracted more so we can evaluate the intensity of the process of annealing the free volume, that is the excess of free volume in each amorphous sample.

The application of the described method enables the immediate reading from the corrected graphs of the free volume change due to any relaxation process. In this way it is easy to obtain the dependence of the free volume change on the relaxation stresses applied to the sample during a definite time. Fig. 3 shows a concrete example of free volume change in dependence on six different stresses acting 300 s on the sample. The first three values were taken directly from fig.2; for reasons of clearness the curves for other stress values were omitted.

Once again, it should be pointed out that the normalization of the values obtained by isothermal dilatometric measurements of amorphous samples leads to a proportionality of the length change and the magnitude of free volume excess.

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