Experimental report

17/02/2016

Proposal: **6-03-425** **Council:** 10/2012

Title: Dynamics and fragility of the metallic alloy NiSi

Research area: Physics

This proposal is a new proposal

Main proposer: **Franz DEMMEL**

Experimental team: Jad KOZAILY
DANIEL NEUVILLE
Louis HENNET
Franz DEMMEL
Margarita FOMINA

Local contacts: Michael Marek KOZA

Samples: Ni1-xSix

Instrument	Requested days	Allocated days	From	To
IN6	4	3	02/08/2013	05/08/2013

Abstract:

Nickel based alloys are important materials with high strength up to high temperatures. Alloying nickel changes various physical properties like strength, liquidus temperature or its solidification behaviour.
The aim of this experiment is to study the relaxation/diffusion dynamics in the binary alloy NiSi towards solidification in the liquid and supercooled state. The aerodynamic levitation set-up from CNRS Orleans for the IN6 spectrometer will be applied. By changing the composition we are able to change the fragility of the alloy. The temperature dependence will deliver the transition temperature from a strong to a fragile liquid. We aim to indentify a slow relaxation process which initiates solidification.

-I- Introduction

Nickel based alloys cover a wide field of applications, for example when high strength at high temperature is needed. To achieve these requirements other elements like aluminium and silicon are added to nickel. For a further optimization of the materials a deeper understanding of the relevant processes towards solidification is necessary. For example, the diffusion coefficient is an important parameter to describe solidification at the liquid-solid interface.

In this experiment, we used quasielastic neutron scattering to determine the Nickel self diffusion coefficients in the $Ni_{75}Si_{25}$ alloys. These data complete an earlier study of the S-rich side of the NiSi phase diagram [1,2].

-II- Experimental details

The high temperature measurements were carried out using the aerodynamic levitation setup specifically designed for neutron diffraction at IN6 and well described by Kozaily et al. [3].

The $Ni_{75}Si_{25}$ spherical samples were made from weighed amounts of pure Nickel and Silicon and then melted in the levitator to spherical shape and homogeneous alloys.

The IN6 spectrometer was operated with an incoming energy Ei=3.12 meV. The chosen incoming wavelength λ=5.12 Å was above the Bragg cutoff of aluminium (from the nozzle).

Inelastic spectra were recorded for about 3-4 hours per temperature. In addition, a background was taken with the empty levitator and argon gas filled sample chamber. A measurement with a vanadium sphere served for detector efficiency corrections and to determine the energy resolution. The measured energy resolution can be described by a Gaussian function with a FWMH = 0.077 meV . The measured spectra were monitor normalized, corrected for detector efficiency including the energy efficiency changes of the Helium detectors and then the measured background has been subtracted.

-III- Measurements

The melting point of $Ni_{75}Si_{25}$ was deduced from the phase diagram as *Tm*=1513 K [4]. Several runs were performed at four different temperatures: 1920 K, 1800 K, 1535 K and 1420 K.

In Figure 1 we show two freezing curves taken at the end of two measurements. Both curves demonstrate that the alloy can be undercooled by up to 250 K for a short time. It can be noted that the run with the lower temperature (T=1420 K) was performed in the undercooled state by about 100 K.

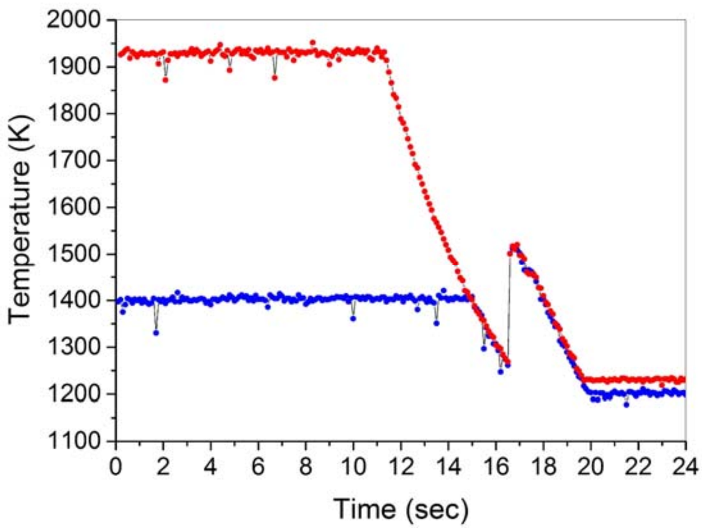

Figure 1. Freezing curves of two runs at the end of the measurement which demonstrate that one run was performed in the undercooled state.

Self-diffusion on long time and length scales can be described with the diffusion equation for the density correlation function of a tagged particle. The spatial and time Fourier transformed solution of the diffusion equation is a single Lorentzian with a Q dependent half width at half maximum (HWHM) $\Gamma_{1/2}(Q)$ [5]:

$$S^S(Q,\omega) = \frac{1}{\pi}\frac{\Gamma_{1/2}(Q)}{\Gamma_{1/2}^2(Q) + \omega^2}$$

At small Q vectors, in the hydrodynamic limit ($Q \rightarrow 0$) a Lorentzian is the exact lineshape for the diffusion behavior and $\Gamma_{1/2}(Q)=DQ^2$. The proportionality constant D is the self-diffusion coefficient for translational diffusion on long distances. To extract the line width a fit with a single Lorentzian function convoluted with the resolution function was applied.

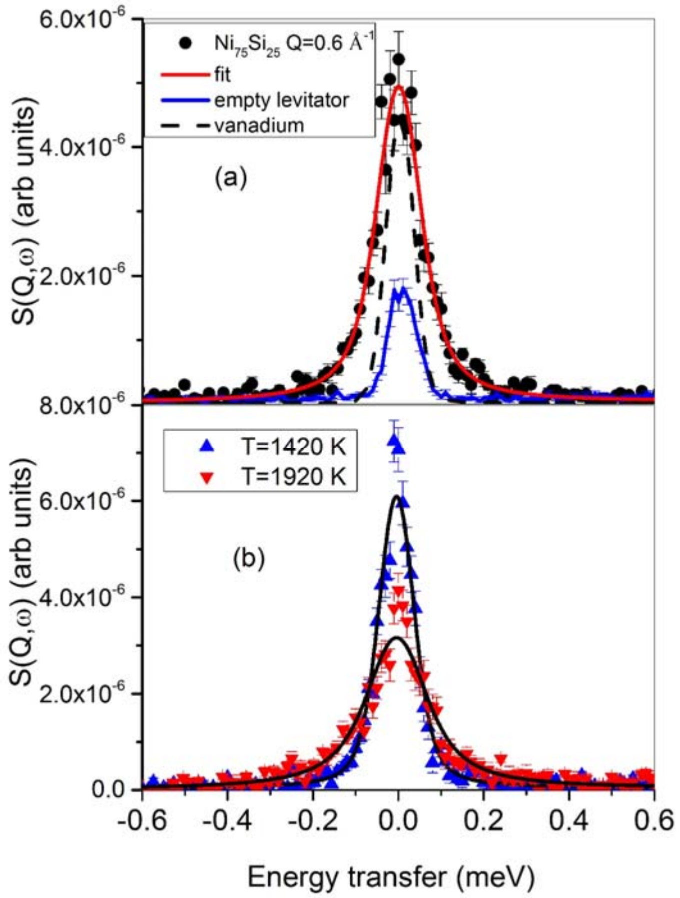

Figure 2. Plot (a) displays a spectrum at Q = 0.6 $Å^{-1}$ and includes measurements with the empty levitator (blue line) and the vanadium sphere (dashed line). Plot (b) shows 2 spectra at Q = 0.5 $Å^{-1}$ for T=1420 K and 1920 K. In both cases, fits with a Lorentzian curve are also presented (red (a) and black (b) lines).

In Figure 2 some spectra including fits with the Lorentzian model are shown. The good fit and the statistics of the data do not recommend the use of a more sophisticated model.

In Figure 2(b) spectra at a wave vector of Q = 0.5 $Å^{-1}$ are shown for the highest and lowest temperatures. For all temperatures the fit with a single Lorentzian function is sufficient and do not indicate a change in lineshape. From the Lorentzian fits we deduced the HWHM which

-IV-Publication

All details of the experiment and the data interpretation are provided in a forthcoming paper:
F. Demmel, L. Hennet, S. Brassamin, D. R. Neuville, J. Kozaily, M. M. Koza, *Nickel self-diffusion in a liquid and undercooled NiSi alloy* (submitted)

-V-References

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