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HIGH PRESSURE POSITRON ANNIHILATION STUDIES OF Fe IN A DIAMOND ANVIL CELL

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Résumé - Nous avons mesuré l'élargissement Doppler du spectre de Fe, jusqu'à la pression de 40 GPa, en utilisant une cellule haute pression à enclumes de diamant (DAC). Cette expérience représente la première réalisation d'une mesure d'annihilation de positrons utilisant la technique des DAC.

Abstract - The Doppler broadening spectrum of Fe has been measured to a pressure of 40 GPa using a high pressure diamond anvil cell (DAC). This represents the first positron annihilation experiment which utilizes DAC technology.

Une goal of high pressure physics is to understand the electronic structure of materials, and how it affects observable physical quantities. An experimental technique both capable of determining the electronic structure of a material and applicable to high pressure studies is the Doppler broadening of positron annihilation radiation. Doppler broadening and angular correlation techniques can provide detailed information concerning electron momentum distribution in a solid, and have become a standard tool for such studies at ambient pressure.1 Although some applications of positron annihilation have lower electron momentum resolution than comparable methods such as de Haas-Van Alphen,2 the positron techniques can be applied to situations which are otherwise intractable.

Some early positron measurements have been made on a few simple metals below 10.0 GPa using Bridgeman anvil devices.3,5 The more recent advent of the diamond anvil cell (DAC) has made experimental studies up to pressures of many tens of gigapascals possible.6 In this experiment, the Doppler broadening spectrum of Fe has been measured to 40.0 GPa.

The Doppler broadening technique was chosen for our first experiment because it allows us to obtain a high count rate, while still having the small sample required to get to high pressures with the diamond cell. Due to the annihilation with electrons of various momenta, the sharp annihilation line at 511 KeV is broadened, with the electron momentum information being contained in the line shape.

The experimental design for the positron annihilation studies is illustrated in Fig. 1. In our experiment, we used a Mao and Bell7 type cell with diamonds which had cutlets of 0.8mm. The sample consisted of a sandwich of two pieces of Fe foil with positron emitting Na22 placed in between. The foils were 99.998% purity and were annealed under an argon atmosphere to remove any defects from the rolling process. The starting sample size was 350 microns in diameter and 100 microns thick. The sample was placed in the hole of a hardened type 301 stainless-steel gasket without any pressure medium. Small ruby chips (5-20 microns) were placed in the sample chamber for pressure
Pressure gradients were measured at various pressures and were found to be at most 3.0 GPa at 40.0 GPa. Errors in the precision of the pressure measurements are estimated to be less than 0.5 GPa at all pressures, based mainly on broadening of the ruby R1 line caused by non-hydrostatic pressures.

This experiment differs from previous positron work at high pressure in that the samples are necessarily small in order to reach 40.0 GPa (0.35mm as opposed to 3-8mm in previous experiments) and the source was deposited directly on the sample foils, rather than on a supporting material. The Doppler broadening data were reduced to the ratio of low momentum events to total events, S. The plot of the S parameter vs. pressure shown in Fig. 2 can be visually divided into three regions. Below the transition pressure, S falls in a continuous manner from the initial pressure of 2.4 GPa to the onset of the bcc (a) to hcp (c) transition at 14.0 GPa. The transition is sluggish and takes approximately 10 GPa to complete. This has been seen from previous x-ray and resistivity data, and is apparent in our positron data as well. From 14 to 24 GPa the Fe appears to be in a two phase region. This region is characterized by a cusp-like feature in the data which is most likely associated with defects, rather than any intrinsic property of the material. It is interesting to note that a similar cusp-like feature is also observed in the positron spectrum of Fe as it is heated through its bcc to fcc transition near 1300K. Above 24.0 GPa, Fe appears to be completely in the hcp phase and the data once more show a smooth decrease of the S parameter with increasing pressure.
The decrease of $S$ with pressure indicates that positrons are annihilating with higher energy electrons at high pressures. This can be understood from the following arguments. The Doppler broadening spectra of metals have been shown to be made up of two components, a valence contribution and a core component. Where the valence contribution goes to zero is the Fermi energy. As the volume decreases, the Fermi energy moves to higher values, which causes the positron annihilation rate with valence electrons of higher energy to increase. In addition, the fraction of annihilations with core electrons increases as the volume is compressed.

An interesting feature of the data is that the rate of change of $S$ with pressure is very different before and after the transition. Conversion of the data to $S$ vs. volume using the P-V data of Mao et al. showed a similar difference, indicating that the data are sensitive to changes in the electron momenta distribution due to other effects, such as the loss of magnetism or changes in the Fermi topology.

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