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Internal Friction Study on C₆₀ Film

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Abstract. We report the study of the internal friction of C₆₀ polycrystal films with different grain size (50 ~500nm) measured at the frequency ranged from 270 to 3140 Hz. At around 260K, an λ -shaped internal friction peak and a modulus drop corresponding to the order-disorder phase transition are observed. With the decrease of grain size, the height of the Q⁻¹ peak and the magnitude of the modulus drop increase. The height of the Q⁻¹ peak is found to be proportional to the measurement frequency. The λ -shaped internal friction peak may be attribute to the fluctuation of new phase embedded in the old phase. The large amount of boundaries in the crystal of little grain size will improve the rate of nucleation of new phase and the peak will become higher. The magnitude of the modulus drop blow 260 K is proportional to the measurement frequency, while above 260 K, the modulus is frequency independent.

1. Introduction

Since the new methods of efficient synthesis of C₆₀ were invented [1], a lot of interesting physical properties of this fascinating material have been reported. Among this properties the structure and phase transition of crystalline C₆₀ are especially attractive. It is now well-known that at room temperature the C₆₀ molecules undergo isotropic rotation around the center of mass of each ball located on a face-central cubic lattice. Around 260 K, the crystal undergoes a first-order phase transition with the four molecules in the conventional fcc lattice becoming orientationally inequivalent, resulting in a simple cubic (sc) lattice with four-molecular basis. In some papers, it is reported that the elastic properties are strongly anharmonic during the phase transition. But their results are quite different. X.D. Shi et al. [2] find for sublimed fcc C₆₀ crystals, the Young's modulus has a 8% jump at 260 K accompanied with a little attenuation peak. S.Hoen et al [3] report that the modulus of single crystal C₆₀ dropped at 269K. A little internal friction peak is also detected. W.Schranz et al. [4] report that the low frequency elastic constant of single-crystal C₆₀ have a negative dip anomaly of 25% at 261K. The elastic properties of polycrystal C₆₀, as reported by N.P.Kobelev et al. [5], is even more different. They found a large internal friction peak and a sharp minimum of the $v_L(T)$ at 250K. So we assumed the different quality of their C₆₀ crystal is a main reason for their different results. We studied the elastic properties and internal friction of C₆₀ polycrystal films and find the grain size of our sample is very important to the variation of Young's modulus and the height of internal friction peak around the order-disorder phase transition temperature.

2. Experiments

The C_{60} we used was prepared using the process described by Kratschmer et al. [1] and purified by column liquid chromatography method to a purity of >99.9%. In a vacuum of 10^{-5} torr, the C_{60} powder was evaporated and deposited on SiO_2 substrates, of dimensions $0.25 \times 4.55 \times 40.00 \text{ mm}^3$. As reported by W.Krakow et al.,[6] we can get a series of samples with different degree of crystallization by varying the growing temperature of our substrates. We carefully control the growing temperature of our substrates at 70°C , 100°C , 200°C , 250°C and get four samples with different grain size. Through the transmission electron microscopy, we could find the degree of crystallization of our samples. As shown in table below:

sample number	growing temperature($^\circ\text{C}$)	average grain size (nm)
1	70°C	100nm
2	100°C	150nm
3	200°C	>400nm
4	250°C	>500nm

The internal friction and the resonance frequency of the composite sample were measured by means of the free-free bar apparatus, in which a combination of electrostatic drive and capacitor microphone detection is used [7]. The internal friction of the composite sample is found to be $\delta = \delta_s + (3t/d)(E_f/E_s)\delta_f$, where t and d represent the thickness of the film and the substrate, δ_f and E_f represent the internal friction and Young's modulus of the film, and δ_s and E_s denote the same quantities for the substrate. The frequencies f of the composite sample are related to the frequencies f_s of the bare substrate by the expression $f^2/f_s^2 = 1 + t/d(3E_f/E_s - \rho_f/\rho_s)$, where ρ_f and ρ_s are the densities of the film and substrate, respectively. A 10^{-2} Torr vacuum is kept in the sample chamber.

3. Results and discussions

We studied the Young's modulus $E_f(T)$ and the internal friction Q^{-1} of our four samples respectively, at the frequency of 1.1 KHz. As shown in fig1a. $\Delta E_f = E_f(T) - E_f(T_0)$.

Fig 1a. The modulus of C_{60} films with different grain size measured at 1.1 kHz.

Fig 1b. The internal friction of C_{60} films with different grain size measured at 1.1kHz.

$E_f(T_0)$ is the Young's modulus of that sample at the room temperature. We can find, of every sample, the ΔE_f has a drop at nd 260K. With the increase of the grain size, the drop become smaller. In fig 1b. the curves of the internal friction Q^{-1} versus temperature of 4 samples are presented. At around 260 K, every curve has a peak and the height of the peak decreases with the increase of grain size. So the degree of crystallization, perhaps the

amount of boundary molecules of our samples play a dominant effects on the magnitude of modulus drop and the height of internal friction peak at the phase-transition temperature.

To study the modulus anomaly and Q^{-1} peak around 260 K further, we do the experiment by varying the measurement frequencies, vibration amplitude and cooling and heating rate. The magnitude of modulus drop and the height of Q^{-1} peak are found to be independent of cooling or heating rate and the vibration amplitude. We find the modulus above 260 K does not depend on frequency. But below 260 K, the modulus for different frequency is quite different. As shown in fig2a. the modulus drop around 260 K is proportional to the frequency. This result indicates the modulus drop, especially the the soft of modulus below 260 K, is not caused by a relaxational process. The mechanism is need to be studied farther.

The height internal friction peak at 260 K is found to be proportional to the frequency as indicated in fig 2b. But it is in no relation of cooling or heating rate and vibration

Fig 2a. The modulus of C_{60} film measured at various frequency. The inset shows the modulus drop varied with frequency.

Fig 2b. The internal friction of C_{60} film measured at two frequencies. The inset shows the height of Q^{-1} peak varied with frequency.

amplitude. Since the phase transition at T_1 (~ 260 K) is of first order, there is a coexistence region, i.e. a temperature range where nuclei of the ordered low temperature phase (short-range ordered clusters) are embedded in the disordered high temperature phase. These nuclei are metastable above transition temperature and can be formally described by order parameter fluctuation, $\delta\eta(r,t) = \eta(r,t) - \eta_0$, where the homogeneous order parameter $\eta_0 = 0$ for $T > T_1$. The fluctuation of order parameter of new phase will give rise to a internal friction peak [4]. But the peak of single crystal is much narrow. As our sample is a polycrystal film, the internal stress will make the transition temperature T_1 and the order parameter fluctuation characteristic lifetime τ_η disperse. So the broader Q^{-1} peak of our sample is understandable. The linear relationship of the height of Q^{-1} peak and the measurement frequency indicated that the fluctuation process with $\omega\tau_\eta \ll 1$ is dominant in our sample.

Since the boundaries and defects in the sample will improve the rate of nucleation, the grain size will certainly play an effect on the internal friction Q^{-1} around the phase transition temperature. With the decrease of the grain size, the amount of boundaries will increase and the Q^{-1} peak will become higher. This is just the case of our result.

In summary, the internal friction peak and the Young's modulus anomaly around 260K are found to be dramatically affected by the grain size of our polycrystal C_{60} film. The linear relation between the height of Q^{-1} peak and the measurement frequency is caused by the fluctuation process of the order parameter of new phase. But the linearly frequency

dependent Young's modulus below the phase transition temperature T_1 can not be explained. The theoretical work should be proceeded further. In single crystal C_{60} , there are a lot of defects (stacking fault defects, boundaries, et al.), which will also affect the Young's modulus and Q^{-1} around the transition temperature. So the different results reported by several papers are understandable.

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