SLOW NEUTRON SCATTERING STUDIES OF LIQUID AND SOLID ARGON

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IDO-17089
AEC Research and Development Report
Physics
TID-4500 (40th Ed.)
Issued: May 1965

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Atomic Energy Division

Contract AT(10-1)-205

Idaho Operations Office

U. S. ATOMIC ENERGY COMMISSION

ABSTRACT

Measurements with the Materials Testing Reactor phased-chopper velocity selector have been made of the slow neutron inelastic scattering from samples of liquid (88.7°K) and solid (82°K) argon. These measurements were made at incident neutron energies of 20, 40, and 100 meV for the liquid samples and at 20, 40, and 60 meV for the solid samples, and spanned a wave vector change κ from 2 to 14 Å⁻¹ for the liquid and from 1 to 11 Å⁻¹ for the solid. The scattering laws for the two phases show distinct momentum dependent coherent inelastic scattering such as has been observed previously in liquid Na and in polycrystalline Be and Al. A frequency spectrum of the normal modes of vibration has been obtained for the solid using the methods of Egelstaff. The spectrum, corrected for multiple phonon excitations, shows two small maxima at 4.7 and 6.9 meV, and has more low frequency modes than either a Debye spectrum or a second nearest neighbor, Born-von Karman spectrum.

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SLOW NEUTRON SCATTERING STUDIES OF LIQUID AND SOLID ARGON

Introduction

The dynamics of liquid argon have recently been studied by Dasannacharya and Rao [1] using a cold neutron spectrometer and a triple-axis spectrometer. They obtained the van Hove self correlation function for times out to 2×10^{-12} seconds and concluded that the width of the self part of the space time correlation function follows a Fick's law for diffusion. Kroo, Brogonovi, Sköld, and Larsson [2, 3] have studied both the liquid and solid phases of argon using a cold neutron spectrometer. From their data they were able to obtain average dispersion relations for both the solid and liquid phases.

The purpose of the experiment reported here was (a) to supplement the above measurements by mapping the complete scattering law over a wide region of energy and momentum transfers and (b) to measure the frequency spectrum of normal modes for the solid. From earlier experiments, it is known that momentum dependent structure at fixed energy transfer can be observed in metallic purely coherent scatterers such as polycrystalline beryllium [4] and aluminum [5]. Also, this momentum dependent structure has been observed in liquid Na [6] which is a partially incoherent metallic scatterer being roughly half coherent and half incoherent. Since argon is nonmetallic and bound only by van der Waals forces, an additional purpose of this experiment was to see if the same type of momentum dependent structure was evident in both the liquid and solid cases.

The Experiment

The experiment was performed on the velocity selector at the Materials Testing Reactor for which a detailed description has been given [7]. Briefly, this velocity selector is a phased-chopper system which produced bursts of monochromatic neutrons at the sample. The neutrons scattered by the sample were detected in banks of one-inch-diameter BF_3 detectors located at 15 scattering angles between 16° and 144° . The energy of the scattered neutrons was determined by time-of-flight methods. The flight path of the scattered neutrons was 2 meters. The detector banks were of graduated width being narrowest at the forward angles. The exact arrangement is given in Harker and Brugger [8], Table II, arrangement II. The angular spread was 3.6° at the forward angles and 7.2° at the largest scattering angle. A fission chamber beam monitor was located in the forward beams 2 meters from the sample.

For the liquid data, incident energies of 0.0198, 0.0396, and 0.0988 eV were used; and the momentum transfer $\hbar\kappa$ spanned the range $2 \leq \kappa \leq 14 \text{ \AA}^{-1}$; while for the solid data, the incident energies were 0.0195, 0.0346, and 0.0591 eV and covered the κ range 1.5 to $10 \text{ \AA}^{-1}$. The chopper speeds were 7500 rpm for the 0.02 eV data and 12,000 rpm for all the higher energy data. The lower speed was necessary at 0.02 eV to maximize the incident intensity for the choppers used. At any given chopper speed, the time width of the burst as measured at the monitor was very nearly a constant, independent of the incident energy. However, the velocity spread in the burst did depend on the incident energy. Table I

shows the velocity selector resolution for the different energies as measured from the standard deviation σ of the burst width at the monitor. The fractional time resolution $\Delta t/t$ is computed from $\frac{2\sigma}{t}$ where t is the time of flight over the 2 meters from sample to the fission monitor. The resolution in energy is given by $\Delta E \cong \frac{2\Delta t}{t} E_0$ and varies from 0.8 to 5.6 meV. No attempt was made to remove the effects of energy resolution, since a systematic reproducible way of accomplishing this has not yet been presented.

TABLE I
VELOCITY SELECTOR RESOLUTION

<u>RPM</u>	<u>E_0 (eV)</u>	<u>2σ (μsec)</u>	<u>$\frac{\Delta t}{t}$</u>	<u>ΔE (eV)</u>
7,500	0.020	21.0	0.020	0.00080
12,000	0.040	13.7	0.019	0.00076
12,000	0.060	12.7	0.021	0.0025
12,000	0.10	13.0	0.057	0.0056

The liquid-nitrogen-cooled dewar used to contain the sample holders was the same one described in Reference 8. The liquid argon was contained in a cylindrical can of 2-1/8 inch diameter and 4-1/2 inches high. The walls were of 0.010 inch aluminum. The sample was liquefied in place and filling was complete when the holder ceased taking gas. The liquid experiment was run at a temperature of $88.7 \pm 1^\circ\text{K}$, and had a measured transmission of 0.86 corresponding to a 10 percent scatterer. The holder for the solid sample was similar but not identical in construction. To reduce the effects of multiple scattering in the sample, cadmium discs of 0.020 inch thickness were placed in the holder 0.75 inch apart and parallel to the plane of scattering. In order to make room for the disc supports, the sample can was made 2.5 inches in diameter. The aluminum wall thickness for this holder was 0.015 inch. The disc supports were cadmium covered and positioned so that their shadow zone was not viewed by the detectors. The solid argon experiment was run at $82 \pm 1^\circ\text{K}$ with a sample transmission of 0.78 corresponding to an approximately 12 percent scatterer.

Treatment of the Data

Data runs from 48 to 96 hours were made with each sample, and during a run a dummy sample holder was cycled alternately with the filled sample holder at ten-minute intervals. The recorded counts were stored in the 8192 channel time-of-flight analyzer, which was operated with 10 μ sec channel widths and 256 channels per angle. The data were then run through a plot-print program. The primary purpose of this program was to examine the quality of the raw data before any processing was done, and to obtain fast background corrections. This program yielded 2 plots for each scattering angle as well as a listing. The plots show the sample data and, superposed on this, the empty sample holder data. Also, the channel-by-channel difference in counting rate was calculated (normalized to the same running times), and averages of their differences over 8 and 16 channels were also plotted. This plot of averaged data, which, of course, shows few details, was used to obtain fast background corrections by normalizing to zero the count rate difference at short and long flight times.

Figure 1A shows a representative plot of this type. The points are the sample counts and the solid line are the open or dummy holder counts. It is

Fig. 1 (A) Raw data -- solid argon. Solid points are for the sample; continuous line is the contribution from backgrounds and the empty sample builder.

(B) Average of the "sample" minus "open" difference. Open circles are an 8 channel average; connected solid points are a 16 channel average. The dotted line is the background correction line obtained from 16 channel average.

for an angle of 41.9° at 0.02 eV incident energy for the solid argon sample. The running time was 2950 minutes each for sample and for the open. The plot scale is such that the highest data points are not shown. Figure 1B shows the average difference in the counting rates. In this the connected solid points are a 16 channel average, and the open points show an 8 channel average. The dotted line drawn on this plot was then used to make a table of fast background connections. This table was used to make the sample-minus-open counting rates zero at large and small flight times.

Using these corrections, the data were converted to partial differential cross section and to scattering law after correction for sample transmission and detector efficiency. The relations used are

$$\frac{d^2\sigma}{dE d\Omega} = \left(\frac{E}{E_0}\right)^{1/2} e^{-\frac{\epsilon}{2}} S_R(\kappa, \hbar\omega)$$

$$\epsilon = \frac{E - E_0}{k_B T} = \frac{\hbar\omega}{k_B T}$$

$$\kappa = \vec{k} - \vec{k}_0$$

and the reduced partial cross section form of the scattering law $S_R(\kappa, \hbar\omega)$ is related to the Egelstaff dimensionless form of the scattering law $S(\alpha, \epsilon)$ [9] by

$$S_R(\kappa, \hbar\omega) = \frac{\sigma_b}{4\pi k_B T} S(\alpha, \epsilon)$$

and

$$\alpha = \frac{\hbar^2 \kappa^2}{2M k_B T} = \frac{1}{A k_B T} [E_0 + E - 2(E_0 E)^{1/2} \cos \theta]$$

where E_0 , E , k_0 , and k are the incident and scattered neutron energy and wave vector, respectively; A is the mass ratio of the target atom to that of the neutron; and θ is the scattering angle.

Since data at more than one incident energy were taken, the scattering law at fixed energy transfer overlaps in regions of κ , and is therefore overdetermined in these regions. Advantage of this overdetermination was taken by averaging the scattering law using the procedure given in Reference 4. This consists of dividing the κ scale into equal increments of $\ln \kappa$, then averaging together all the scattering law points of a given energy transfer that lie in such an increment. Figure 2 shows representative results of such a procedure for solid argon at an $\epsilon = 0.4$. The solid points are the data, and the open circles are the results of averaging. Generally, 20 adjacent increments of κ were chosen to span the range of the data, and include in some cases up to 120 data points. This figure also clearly shows the presence of momentum dependent structure in the scattering.

Fig. 2 Reduced partial differential cross section for solid argon at $\epsilon = 0.4$ illustrating the effect of averaging the data over small increments of $\ln \kappa$.

Results: Solid Argon

Figure 3 shows some representative reduced partial differential cross sections for solid argon at fixed scattering angles. These are for the 0.02 eV incident energy and are shown as $S_R(k, \hbar\omega)$ versus ϵ . The contributions due to coherent inelastic scattering, though small, show up quite clearly on either side of the elastic peak in spite of the partial incoherence of the sample. ϵ has been defined for the solid data in terms of the melting temperature (83.9°K) so that $\epsilon = 1$ corresponds to 0.00723 eV. Multiple phonon effects are clearly present, as is evidenced by the relatively high inelastic cross section for $\epsilon > 1$, ie, for excitations larger than $k_B T_M$ where anharmonic forces that give rise to multiphonon events are present. It is especially noticeable for the angles 71.7° and 95.5°. Though part of this is undoubtedly incoherent scattering, most of the cross section arises from coherent effects. In similar cross sections taken at the higher incident energies, such multiple phonon effects are even more pronounced.

Figure 4 shows the elastic or zero energy transfer scattering law for solid argon. The different symbols correspond to runs of different incident energy. The slash marks at the top show the Bragg peak positions. To within the angular resolution of the apparatus, the scattering law clearly shows most of the expected Bragg peaks. Since the width of a Bragg peak at a fixed scattering

Fig. 3 Representative cross sections at fixed scattering angle for solid argon for an incident energy of 0.02 eV.

Fig. 4 Reduced partial cross section for solid argon for elastic scattering. The slash marks indicate positions of expected Bragg peaks.

angle is very nearly the same as that of the burst width, the same number of channels were summed over as in the burst and assigned to the elastic scattering.

Figure 5 shows the scattering law for solid argon. These plots are in the form of reduced partial cross section at fixed values of the energy transfer plotted as a function of the wave vector change k . The plots are all composed of data averaged in the way mentioned above. The vertical slash marks again give the k values for Bragg scattering. The solid curves are drawn only to guide the eye. The presence of momentum dependent structure is quite clear, and exists out to energy transfers of $\epsilon \lesssim 0.7$. The peaks in this structure at fixed ϵ are centered around the Bragg values of k . Further, no shift of the structure with increasing k is evident; the structure merely becomes smeared out. This lack of shift in the structure indicated that at the high frequency end, to within the resolution of the equipment, the contributions of the longitudinal branch of

Fig. 5 Scattering law for solid argon.

the dispersion relation are not well separated from those of the transverse branches. In contrast, for aluminum [5], which has the same structure as argon (fcc), such a separation of longitudinal and transverse branches exists and causes a shift of the structure in the scattering law.

Liquid Data

The data for liquid argon were treated in the same fashion as the solid data. Figure 6 shows the scattering law for the liquid at 88.7°K. Again momentum dependent structure is noted in the inelastic scattering such as has been observed in liquid sodium [6]. The structure, however, is not as sharp as that of the solid, which is what might be expected in a liquid, though again it appears to exist out to energy transfers of $\epsilon \lesssim 0.7$. Except for the smearing out of the structure, the data are quite similar to that of the solid and again there is no apparent shift in the structure with increasing ϵ . No attempt has been made to obtain a frequency spectrum for the liquid since the poorer energy resolution (0.0056 eV) obtained at the relatively high incident energy of 0.1 eV would prevent seeing any details at all in the spectrum. Also no attempt has been made to obtain average dispersion relations in the manner of Kroo et al [2,3].

Frequency Spectrum for Solid Argon

Since argon is one of the simplest materials and is bound in a solid only by weak van der Waals forces, its frequency spectrum of normal modes is of interest. The Egelstaff extrapolation procedure [9] was used in attempting to obtain a frequency spectrum. This method involves extrapolating curves of $S(\alpha, \epsilon)/\alpha$ from a region of relatively large α to $\alpha = 0$. This extrapolation defines a function $p(\epsilon)$ given by [9]

$$p(\epsilon) = \epsilon^2 \lim_{\alpha \rightarrow 0} S(\alpha, \epsilon)/\alpha$$

and the frequency spectrum of normal modes $\rho(\epsilon)$ is related to $p(\epsilon)$ by

$$\rho(\epsilon) = \frac{2}{\epsilon} \sinh(\epsilon/2) p(\epsilon) .$$

The method is based on the assumption that only harmonic forces are important, and secondly that the incoherent approximation to the scattering is valid. It has been primarily applied to incoherent scatterers such as water [10] and vanadium [3], but has also been used for such purely coherent scatterers as beryllium and beryllium oxide [11]. Since argon is partially incoherent ($\sigma_{inc} = 0.18b$, $\sigma_{coh} = 0.5b$) [12], the method may be applicable. However, for argon which has a Debye temperature of only 80°K (or for that matter any other material having a low Debye temperature), the extrapolation method is not well suited for obtaining a frequency spectrum. The difficulty does not lie so much in the validity of the method itself, but rather in its application, and only limited success can be expected in obtaining a spectrum. The reasons are as follows: In order to do the extrapolation of S/α to $\alpha \rightarrow 0$ in such a partially coherent scatterer, extrapolation must be made from a region of α values that are not too large, yet are sufficiently large that they lie beyond the region of coherent effects. In argon, coherent effects, which show up as oscillations in the scattering law, exist out to $\kappa = 7 \text{ \AA}^{-1}$, or $\alpha = 0.3$. Now the one phonon frequency

Fig. 6 Scattering law for liquid argon.

spectrum for monoatomic material must go to zero at energies not larger than that corresponding to the melting temperature, and will usually vanish for values much less than this. In argon, the Debye temperature is quite close to the melting temperature which has an energy $k_B T_M = 0.00723$ eV. Hence, in order to measure a spectrum of which the total range is only of the order of 0.007 eV, cold neutrons would be preferred since they would give the best energy resolution and thus be the most sensitive to the shape of the frequency spectrum. However, this cannot be done because cold neutrons do not give α values large enough to lie beyond the coherent region of the scattering law. Therefore, higher incident neutron energies must be used to reach the appropriate α ranges. The price paid, however, is a poorer energy resolution and, thus, a loss in sensitivity in determining this frequency spectrum. The problem is further complicated by the fact that incident energies sufficiently large to yield data in the correct region of α are from 3 to 8 times larger than $k_B T_M$. Consequently, multiple phonon effects are quite prominent and tend to obscure the single phonon effects. These effects in argon are so large that the frequency spectrum obtained by extrapolation only begins to converge to 0 at energies beyond $3 k_B T_M$. Hence, the energy of the upper limit of the true one phonon spectrum, which should not exceed $k_B T_M$, will not be determined reliably by the extrapolation, but must be assigned on some other basis. Also, as is shown in Figures 8 and 9, the slopes of the computed S/α curves at large α differ by very little in spite of the fact that the frequency spectra used differ quite markedly among themselves.

The method used in this experiment is to assign arbitrarily the cutoff frequency to be near the melting point and then to try various shapes for the frequency spectrum. Having assumed a function $p(\beta)$, this function is used in the LEAP program where multiphonon contributions are computed, and the output results of $S(\alpha, \beta)/\alpha$ versus α are compared with the data plots of S/α . Based on these comparisons, a new $p(\beta)$ is estimated. This process was repeated until a satisfactory fit of the computed curves to the data was obtained.

In deciding whether the computed curves agree with the data, the data for α values below 0.2 were ignored for two reasons. First, the coherent oscillations in the S/α data make it difficult to decide when a fit has been made. Secondly and more important, for the small α values, multiple scattering effects are quite pronounced. They tend to steepen the slope of the data plots. The region in α where such effects are important can be estimated conveniently by measuring the first moment of the energy transfer of the scattering law [6]. Figure 7 shows the result for solid argon. For κ values $< 5 \text{ \AA}^{-1}$ ($\alpha < 0.2$), the first moment is higher than predicted. This is caused largely by multiple scattering in the sample. The Cd spacers in the sample are of some help in reducing multiple scattering, but do not completely eliminate it.

Figure 8 shows representative data plots of $S(\alpha, \epsilon)/\alpha$ versus α for various

Fig. 7 Ratio of the observed to the calculated first moment of the energy transfer for solid argon as a function of κ .

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Fig. 8 Comparison of solid argon data to results calculated by LEAP from assumed frequency distributions.

values of ϵ . Also shown are smooth curves obtained from the LEAP program for different assumed forms of the frequency spectrum. The solid curves are the results of repeated fitting of the spectrum and represent the best overall fit. For comparison, curves corresponding to a simple Debye spectrum, $\theta_D = 81.9^\circ\text{K}$, are also shown as are the curves resulting from using the Born-von Karman spectrum as calculated by Kroo et al [2, 3]. The frequency spectra $\rho(\epsilon)$ for these are shown in Figure 9. The spectrum obtained by Kroo et al was calculated using the Born-von Karman theory with a second nearest neighbor central force model. The force constants used by Kroo et al were obtained directly from an assumed interatomic potential (Buckingham-Rice potential). This was done since the single crystal elastic constants are not known for argon. The resulting frequency distribution has a cutoff at $\epsilon = 1.1$ which is slightly above the melting temperature. It is included here since it is the only Born-von Karman theoretical curve available.

Fig. 9 Various frequency spectra assumed for solid argon. The solid curve gives the best overall fit.

It is seen that the spectrum obtained from the experiment has a shape roughly similar to the Born-von Karman shape, though the high energy peak lies at a slightly lower energy. It has more low energy contributions than either the Debye or Born-von Karman spectrum, and less contributions at the high energies. The shapes of the two maxima in the experimental spectrum cannot be determined in detail since the energy resolution is not sufficiently good.

Conclusion

Scattering laws for liquid and solid argon have been measured. These scattering laws show the same kind of coherent inelastic effects in the liquid that have also been observed in liquid sodium. Similarly, these same effects occur in the solid. The one phonon frequency spectrum has been obtained for the solid and shows some evidence of structure. However, because of the low Debye temperature, the instrumental energy resolution cannot resolve more details of the shape. This experiment also indicates for such materials that the extrapolation method of obtaining a frequency spectrum is unreliable.

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