A general purpose cold finger using a vibration-free mounted He closed-cycle cryostat[a)]

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A method for mounting a He closed-cycle cryostat which consists of an exchange gas envelope around the cold head to cool an independently supported sample mount as in model DE202 with a DMX-20 interface from APD Cryogenics, Inc. is described. No detectable vibration of the sample mount is observed as evaluated using ^{57}Fe Mössbauer spectroscopy and a piezoelectric accelerometer. Using a 25 μm thick α-Fe foil a linewidth of 0.231(3) mm/s at 300 K with the refrigerator on and the same linewidth with the refrigerator off is observed. The easy optical access afforded by such a cold finger makes it an economical general purpose laboratory tool for performing low-T spectroscopic investigations, such as microwave, optical, γ-ray, x-ray and neutron-scattering measurements. Other applications include electrical transport, SIMS, RBS, and rare-gas matrix isolation. © *1995 American Institute of Physics.*

I. INTRODUCTION

During the past decades, commercially available He closed-cycle refrigerators (CCR), based mostly on the Gifford–McMahon cycle, that can routinely cool to 10 K or lower and run continuously without the need for liquid cryogens have attracted widespread interest. In institutions around the globe where liquid helium is not available, CCRs offer the only means to perform low-T measurements for scientific and/or technological missions. On the other hand, in institutions where liquid helium is readily available, use of CCRs offers substantial savings in supply costs and operational efficiency. These derive in part from the costs of liquid cryogens (helium and nitrogen) and storage vessels, and in part from the convenience of not having to periodically replenish a research vessel at odd hours. In spite of these advantages, CCRs have found limited use in low-T measurements up to now. There are several reasons for this.

First, a problem arises from the operation of these refrigerators, viz., mechanical vibrations intrinsic to the expander. These mechanical vibrations are transmitted to the sample through the cold head. In some types of physical measurements this is acceptable, but in others the vibrations are fatal, as illustrated in Table I. This work is primarily concerned with applications where even traces of mechanical vibrations on the cooled sample totally defeat the use of CCRs.

Broadly speaking, two means have been employed to provide thermal coupling while mechanically decoupling the vibrating cold head from the sample. One of these is to use a Cu-finger stock or braids, and the other is to use a He exchange gas envelope around the cold head. In both cases, independent mechanical support of the sample holder assembly is provided. In the present work, we have used the latter approach employing a commercially available CCR. We have developed a mounting system for the Displex model DE202 He closed-cycle-refrigerator[1] with a DMX-20 interface made by APD Cryogenics, Inc., Allentown, PA. We have evaluated it for mechanical vibrations using a conventional piezoelectric accelerometer and, independently, a more sophisticated probe of vibrations—Mössbauer spectroscopy. In the latter, the presence of instrumental vibrations are manifested as broadening of the nuclear resonance, which can be studied as a function of temperature noninvasively. The Al vacuum shroud of the DMX-20 poses difficulties in use, as discussed in Sec. II. We have made a modification of the shroud which not only facilitates Mössbauer spectroscopy applications, but is indispensable for other applications requiring vibration-free operation as we will discuss later. These innovations are incorporated in a mounting system that is now commercially available[2] from Nomel Technologies, Inc. of Cincinnati, OH. Over the years we have routinely used such a system in ^{57}Fe, ^{119}Sn, and ^{125}Te Mössbauer spectroscopy measurements on high-T_c superconductors[3] and chalcogenide glasses[4,5] particularly in T-dependent measurements with consistently reliable results.

II. CLOSED-CYCLE REFRIGERATOR CONFIGURATION AND MOUNTING SYSTEMS

We purchased a Displex model DE202 CCR with a model DMX-20 vibration isolation interface from APD Cryogenics, Inc.[1,6] In order to take full advantage of the vibration isolation feature of this refrigerator/interface combination, it was first necessary to independently suspend the vibrating cold head (expander module) and the DMX-20 interface with sample attached (see Fig. 1). This was accomplished by hanging the expander module from a rigid tower, itself attached to a concrete laboratory ceiling. The tower was designed to allow a limited height adjustment for refinement of vertical alignments, if necessary. Added cross-bracing of this tower was necessary to ensure rigidity was not compromised, since one of the tower's functions was to faithfully transmit cold-head vibrations to the surrounding

[a)]U.S. Patent No. 5,327,733, issued on July 12, 1994.
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TABLE I. Selected applications of closed-cycle refrigerator for measurements in the physical sciences and their vibration-free requirements.

Applications	Vibration-free requirement
Light scattering (Raman, photoluminescence, infrared)	not stringent
Micro-Raman and microphotoluminescence	very stringent
X-ray diffraction	very stringent
Neutron scattering	not stringent
EXAFS	not stringent
Mössbauer spectroscopy (transmission, conversion electron)	very stringent
Rare-gas matrix isolation—Mössbauer	very stringent
Electrical transport measurements	stringent

structure. The DMX-20 interface was supported by an open trapezoidal framework of aluminum struts attached to a massive, sand-filled table. The sand-filled table was used not only for its mass, but also because the loose-packed sand provides an effective vibration-damping medium. Both mass and vibration damping are necessary to ensure rigid and vibrationless support of the DMX-20 interface and the sample attached to it.

Once in place, horizontal alignment of the cold head and DMX-20 interface is crucial to eliminating vibration. Some of the clearances between heat exchangers and the DMX-20 interface are quite small (at most a few millimeters), so centering the cold head inside the interface and maintaining that centered position is critical to the success of the mounting system. The alignment is obtained by using spacers about 1/2 in. in thickness and securely tightening the four screws provided, mating the cold-head flange with the interface flange. In order to increase counting rates, the Mössbauer source–detector distance was reduced as far as possible. We developed a recessed window to project the source in the vacuum shroud. The limitations imposed by the single-piece shroud and radiation shield required more extensive modifications as we discuss next.

When we mounted the refrigerator, the base of the vacuum shroud was placed approximately 15 cm above the table top. This distance was chosen because it was convenient to place the centerline of the Mössbauer transducer in line with the center of the sample about 18 cm above the table. To remove the single-piece vacuum shroud and radiation shield for either sample exchange or maintenance, we made provision for a hole in the center of the massive table as illustrated in Fig. 1. However, every time a sample was changed, the transducer assembly had to be decoupled, and then recoupled and realigned with the vacuum shroud. To reduce the time and effort needed to change a sample and to keep the alignment of the transducer in relation to the shroud intact, we replaced the one-piece Al shroud and radiation shield by a two-piece vacuum shroud and divided the radiation shield into two pieces. The upper half of the vacuum shroud and radiation shield was mechanically anchored to the DMX-20 interface *at all times*, while the bottom half could be removed for easy access to the sample mount. A

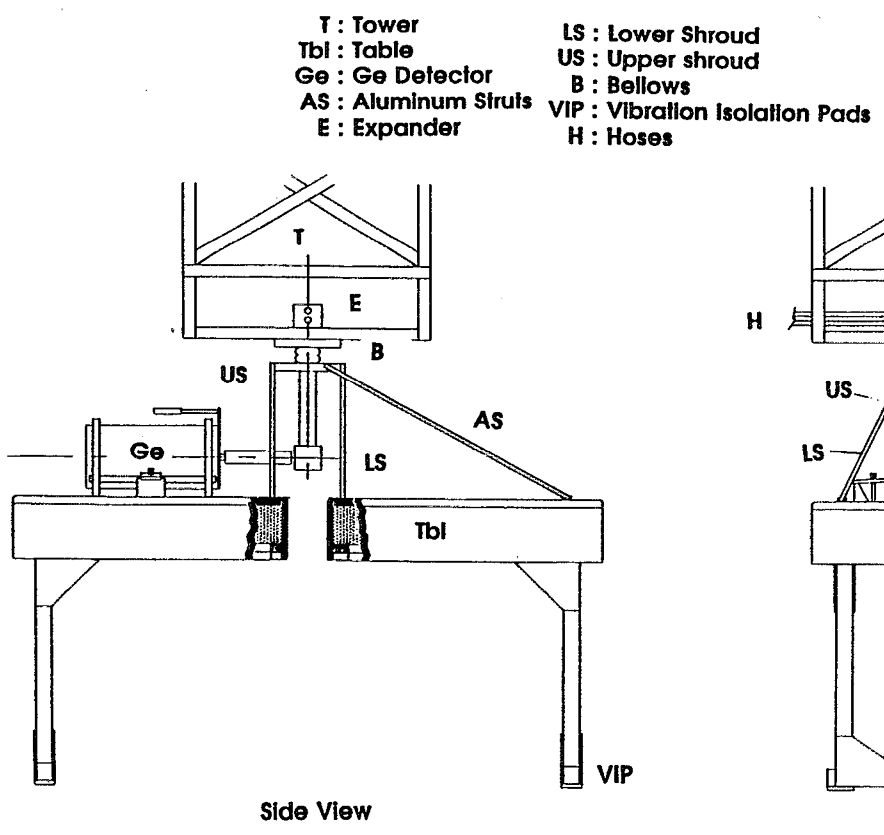

FIG. 1. Schematics of mounting method. The cold head is supported by a rigid tower from the ceiling while the DMX-20 interface is positioned on top of a heavy sandbox with the aid of aluminum struts. The hole in the table permits servicing of the cold head.

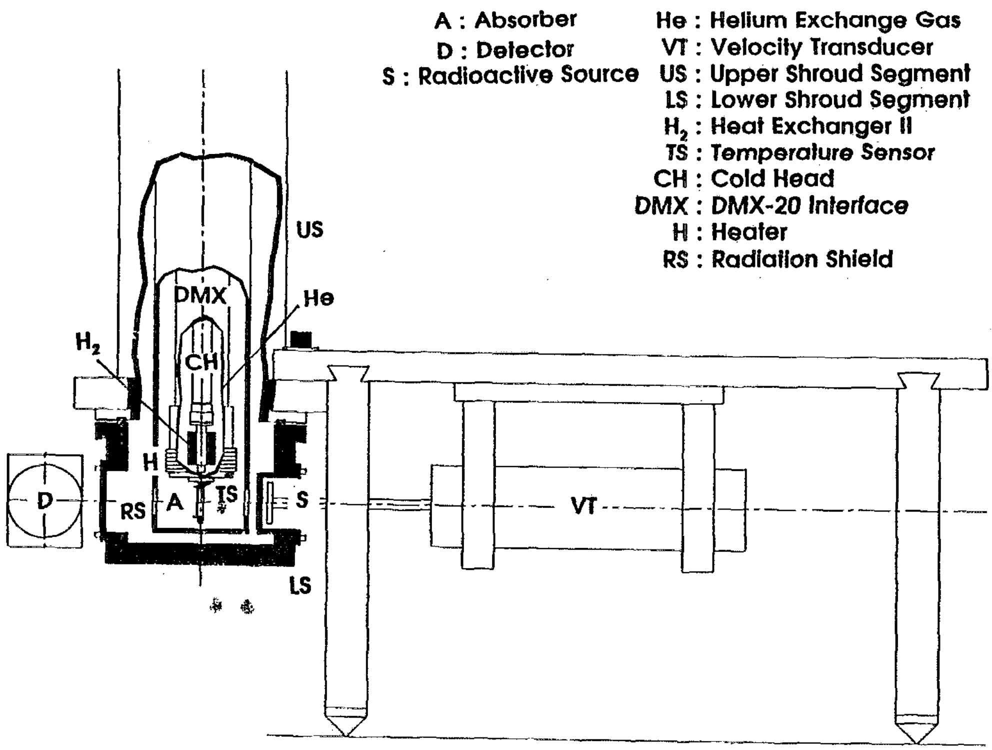

FIG. 2. Schematics of split vacuum shroud and Mössbauer transducer assembly. Note that mechanical coupling of the Mössbauer transducer (T) assembly to the upper segment (US) of shroud once made is held intact. Sample A is accessed by translating transducer back on its track, dropping the lower segment (LS) of the shroud by breaking the vacuum and unscrewing the radiation shield.

key feature of our two-piece vacuum shroud design is the complete absence of a traditional vacuum flange. The tail of the shroud is held in place by vacuum (as shown in Fig. 2), without any need to tighten bolts or other fasteners in order to obtain and maintain a seal. This also eliminates the possibility of improperly tightening and warping a flange.

The last requirement of a vibration-free system is a rigid coupling between the DMX-20 interface and the spectrometer (in the case of Mössbauer spectroscopy, the transducer). The transducer is placed in a support assembly rigidly attached to the top half of the two-piece vacuum shroud. In this way, the transducer assembly always remains mechanically coupled (its alignment assured) even while changing samples. The transducer could be displaced to and fro and locked in position in a track at any point in its support assembly. We found it necessary to allow the transducer support assembly in some sense to "float" on the table top, but always rigidly attached to the DMX-20 interface. This minimized unwanted relative motion between the transducer support assembly and sample, leading to the narrowest linewidths in Mössbauer effect measurements as discussed in Sec. IV.

III. EVALUATION OF SYSTEM PERFORMANCE BY PIEZOELECTRIC TRANSDUCER

We used a Hewlett–Packard model 35670A four-channel digital signal analyzer with a PCB 339A11 triaxial piezoelectric transducer made by PCB Piezotronics, Inc. Depew, NY, 14043-2495, to evaluate the vibration-free performance of the mounting system. The piezoelectric accelerometer (transducer) was mounted directly on the sample mount with some wax. Tests were conducted at room temperature only, first with the expander off and then on. Time spectra were recorded in 2048 channels, with a scan lasting 2 s. Typically, a total of 20 scans were taken to average the information for one spectrum. Fourier transforms of the time spectra yielded the frequency responses of the acceleration experienced by the piezoelectric transducer and these are displayed in Fig. 3. Results shown in Fig. 3(a) were obtained at ambient conditions with the expander off while those shown in Fig. 3(b) were obtained with the expander on.

Several observations become clear from the results displayed in Fig. 3. The typical magnitude of acceleration experienced by the transducer is less than 10^{-10} g at ambient conditions [Fig. 3(a)], and it represents the sensitivity resolution of the piezoelectric transducer used. When the cold head is aligned with the interface, no discernable change in acceleration of the piezoelectric transducer is observed upon switching on the compressor. In general, these results amply demonstrate the vibration-free nature of the mounting system developed by us.

IV. EVALUATION OF SYSTEM PERFORMANCE BY MÖSSBAUER SPECTROSCOPY

The intrinsically narrow natural linewidth ($2\Gamma_n=0.196$ mm/s) of the 14.4 keV γ resonance provides a convenient method for ascertaining instrumental broadening due to residual vibrations in a Mössbauer spectrometer. Often, the

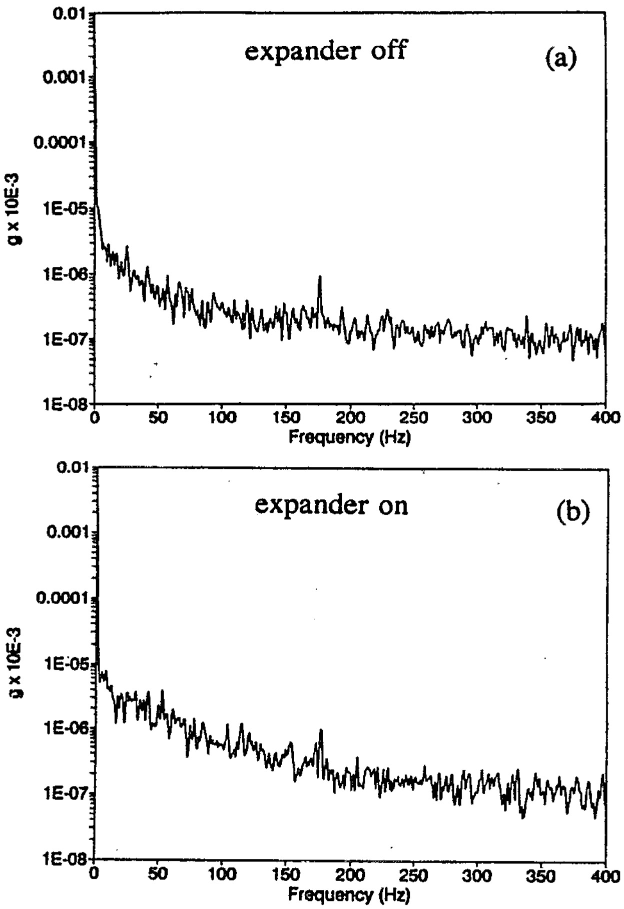

FIG. 3. Response of piezoelectric accelerometer (mounted on sample holder) along the source–absorber axis, when (a) the expander is off, and (b) the expander is on. Note that when the cold head and interface are aligned, the accelerometer response is unaffected upon switching on the expander.

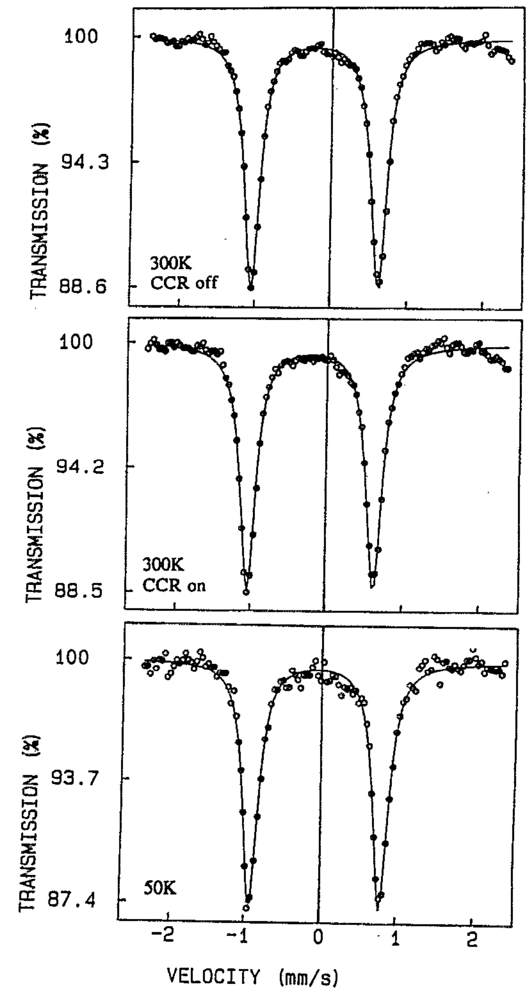

FIG. 4. Spectra of the inner two lines of an α-Fe taken with a ^{57}Co/Pd emitter when the absorber is (a) at 300 K with CCR off, (b) at 300 K with CCR on, and (c) at 50 K with CCR on. Note that upon cooling (50 K), the splitting between the two lines increases as the internal field increases, and the centroid of the two lines moves towards positive velocities because of the second-order Doppler shift. One observes no change in linewidth between spectrum (a) and (b), clearly indicating the vibration-free nature of the system mount

observed linewidth on the inner two lines of the six-line magnetic hyperfine structure of α-Fe is used as a critical measure of spectrometer performance.

A commercially purchased 10 mCi ^{57}Co source in Rh metal and a second ^{57}Co source in Pd metal were used to excite the 14.4 keV nuclear resonance in a 25 μm thick Marz grade pure α-Fe foil from Materials Research Corporation, NY. The Mössbauer spectrometer consisted of a 256 channel analyzer using a PCA-II board from Oxford Instruments, Inc. with a 286-based microcomputer and a constant acceleration drive from Austin Science Associates. The spectra were least-squares fit to a Lorentzian line shape keeping location, width, and intensity as variables. Gamma rays were detected with a Kr-filled proportional counter.

The sample temperature was controlled to ± 0.050 K in the range 12 K$<T<$300 K, using a pair of Si diodes and a cryogenic temperature controller. Spectra were accumulated systematically as a function of temperature in the range 12 K$<T<$300 K. Spectra were also recorded at T=300 K with the CCR on, and then off. The T-dependent measurements were performed to ascertain if there are any sources of vibration intrinsic to the operation of the cryostat at low T. In a properly "tuned" mount of the cold head, we could not detect any line broadening that could be ascribed to vibration. Aligning and tuning the system involves a series of crucial steps performed in a certain order. It includes (a) alignment of the expander and interface, (b) a flush of the exchange gas envelope to completely remove traces of air and water vapor, (c) mounting of the absorber in the holder so that it will not loosen due to thermal contraction and/or develop temperature gradients upon cooling due to lack of adequate thermal contact, (d) dampening of the vibration from the pumping system transmitted to the shroud through the vacuum line, and finally, (e) providing a firm mechanical coupling of the expander assembly to the tower to carry expander vibrations away from the table supporting the interface and absorber.

A. RESULTS

Typical spectra obtained in the present work are shown in Fig. 4. Figure 5 displays results of the present measure-

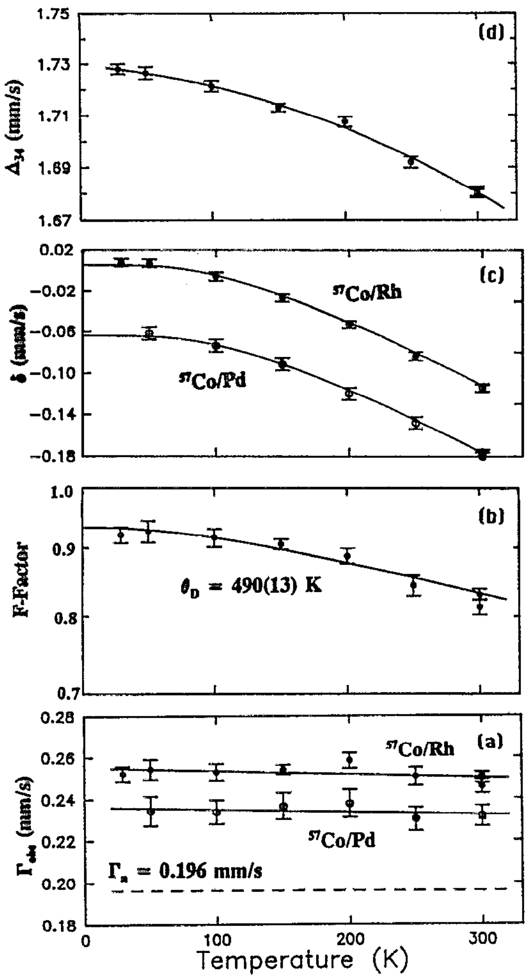

FIG. 5. T dependence of Mössbauer effect parameters of α-Fe: (a) observed linewidth, (b) Lamb–Mössbauer factor, (c) isomer shift, and (d) splitting of the inner two lines. Measurements with a $^{57}Co/Rh$ (filled circles) and independently with a $^{57}Co/Pd$ emitter (open squares) were made.

ments on α-Fe. The linewidth (FWHM) observed using the $^{57}Co/Pd$ source with an α-Fe foil at 300 K, with the expander off, was found to be 0.231(3) mm/s. This linewidth will henceforth be denoted as the base linewidth (Γ_b) for comparison with results obtained after switching on the expander.

At the outset we recognize that the base linewidth is largely determined by the intrinsic emission linewidth of the $^{57}Co/Pd$ source (Γ_s), intrinsic linewidth of the absorber (Γ_a) foil, and linearity of the constant acceleration drive. A least-squares fit of the lineshape to a doublet gave a 12% absorption effect with the 25 μm thick Fe foil, and we could achieve a statistical uncertainty amounting typically to less than 2% of the observed linewidth, i.e., 0.004 mm/s. A linewidth measurement performed after switching on the expander, and letting the system thermally equilibrate at 300 K, yielded a value of 0.231(3) mm/s, identical to the base linewidth value. No changes in the drive or error signal of the K4 motor could be detected on an oscilloscope upon switching on the compressor.

Next, the absorber temperature was lowered in steps of 50 K, and spectra systematically recorded at several temperatures in the 12 K$<T<$300 K interval. The results of these measurements are summarized in Fig. 5. Parallel measurements were also performed with a $^{57}Co/Rh$ source using the same α-Fe absorber and the results with this source are also included in Fig. 5.

1. Observed linewidths

At the outset we note from Fig. 5(a) that a somewhat larger base linewidth [0.255(3) mm/s] is observed with the $^{57}Co/Rh$ source than with the $^{57}Co/Pd$ source [0.231(3) mm/s] at room temperature. This systematic effect represents the intrinsically broader emission linewidth of the $^{57}Co/Rh$ source (Γ_s) in relation to the $^{57}Co/Pd$ source. Furthermore, with both sources, we find the linewidth to systematically increase by about 0.004 mm/s upon cooling the α-Fe foil from 300 to 10 K. As shown in the Appendix, this small increase in linewidth can be completely accounted for by an increased effective thickness ($x=n\sigma_0 f$), where n is the number of ^{57}Fe nuclei/cm^2, σ_0 the maximum resonant cross section, and f the recoil-free fraction of the α-Fe foil due to an increase in the recoil-free fraction upon cooling. One is thus led to conclude that no line broadening observed at low T can be attributed to vibrations intrinsic to the operation of the CCR expander.

2. Recoil-free fraction

The integrated area under the inner two lines of α-Fe studied as a function of temperature was found to systematically increase upon cooling [see Fig. 5(b)]. This is due to an increase in the recoil-free fraction of α-Fe upon cooling. We have fit the observed T variation to a Debye vibrational density of states and obtain a θ_D=493±13 K. The present θ_D result for α-Fe is in good accord with previous values reported.[7]

3. Isomer shift

The displacement of the centroid of the inner two lines of α-Fe from zero velocity provides the isomer shift of the emitter matrix relative to the standard absorber. Upon cooling the α-Fe foil, the isomer shift becomes positive as can be seen from comparing the spectra at 300 K with the one at 15 K, and this is due to the second-order Doppler shift. At room temperature the isomer shifts of Rh and Pd metal relative to α-Fe were fixed, respectively, at −0.177(2) and −0.114(2) mm/s from previous work.[8] The T dependence of the isomer shift $\delta(T)$ for α-Fe measured using the source in the Rh-matrix and the one in Pd-matrix display [Fig. 5(c)] a behavior that is characteristic of the second-order Doppler shift. We note that the shift between the Rh and Pd matrix remains nearly constant at 0.061 mm/s as one would expect, since in these experiments the sources were always kept outside the cryostat at 300 K.

4. Magnetic splitting

The splitting [Fig. 5(d)] between the inner two lines of α-Fe studied systematically as a function of T increases upon cooling the absorber. This is due to an increased magnetic hyperfine field at Fe nuclei in α-Fe upon cooling due to

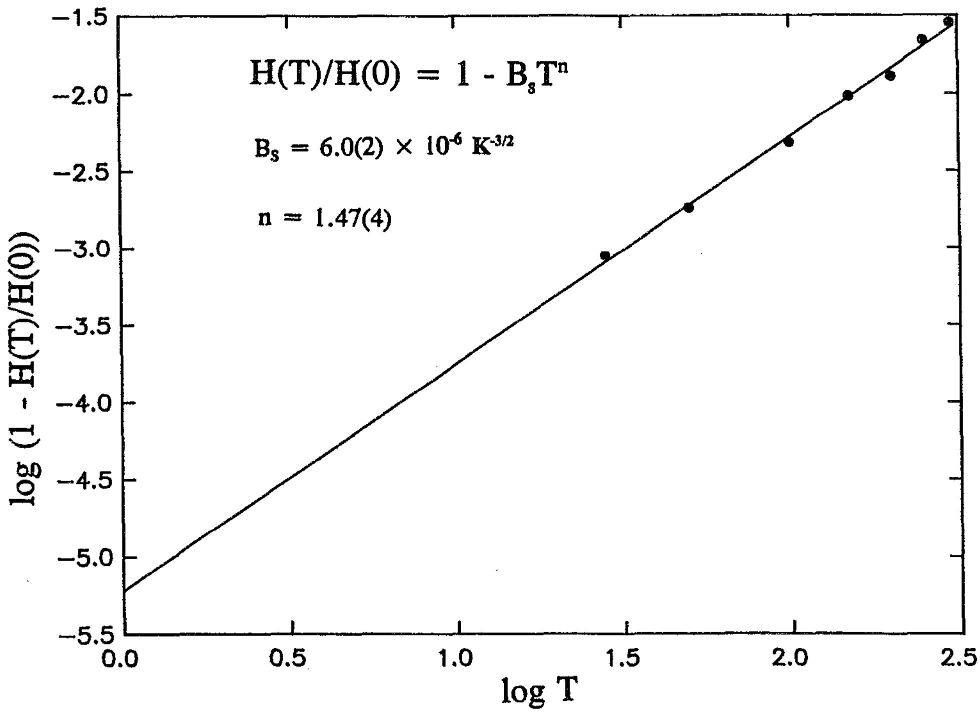

FIG. 6. Results of Fig. 5(a) for α-Fe replotted as a log of $[1-\Delta(T)]/\Delta(0)$ vs log T displaying a $T^{3/2}$ behavior characteristic of magnon excitations.

spin-wave excitations (magnons). As discussed elsewhere one expects the hyperfine field to display a $T^{3/2}$ dependence. We have fit the observed variation in hyperfine field $H(T)=H_0(1-B_sT^n)$ and find the stiffness parameter $B_s=6.0(2)\times10^{-6}\,K^{-3/2}$ and the power law $n=1.47\pm0.04$ as shown in Fig. 6. Our values of B_s and n for α-Fe compare favorably to those reported earlier ($B_s=5.2\times10^{-6}$ and $n=3/2$) by Guitierrez *et al.*[9]

B. Comparison of system performance with previous work

The earliest groups to use CCRs for Mössbauer spectroscopy work were those at Columbia University[10] and at Johns Hopkins University,[11] to the best of our knowledge. Formally, although no linewidth result is quoted in their paper,[10] the group at Columbia did provide spectra of α-Fe taken with the CCR on, and also with the CCR off. A cursive examination of their spectrum (Fig. 2 in Ref. 10) shows that the size of the effect on the outermost lines (lines 1 and 6) changed from 23.8% when the CCR was off, to 19.5% when the CCR was on. If both measurements are performed at the same temperature and used the same absorber, which we think is likely, then the 18% reduction (23.8%→19.5%) in intensity indicates at least a line broadening of the same amount due to the expander motion.

Pfeiffer *et al.*,[12] and independently Rosov *et al.*,[13] have used a Displex DE202 with a DMX-20 interface from APD, Cryogenics, Inc. for Mössbauer spectroscopy work. Neither group has reported a linewidth measured using α-Fe, so that a quantitative comparison is not feasible.

Alonzo *et al.*[14] have used a Gifford–McMahon CCR model K1, from Officine Galileo, Florence, Italy, and adapted it for Mössbauer spectroscopy work. Cu braids were used to cool the source and absorber and to decouple vibrations of the cold finger resulting from expander motion. In their system, the authors report observing 20% line broadening on the six-line spectrum of α-Fe due to expander motion. Baldini *et al.*[15] developed a variation on the mounting setup of Alonzo *et al.*[14] and achieved a lower operating temperature (10 K) than Alonzo *et al.* could (30 K), but the observed linewidth did not appear to be any narrower.

CRYO Industries of America, Inc. in Atkinson, NH 03811 have recently announced[16] a He CCR model REF-399-D22 for Mössbauer spectroscopy applications. Although no specific linewidth values and mounting details are currently available, a line broadening of less than 10% on the inner two lines of α-Fe has been claimed. A similar He CCR system is marketed by Janis Research Company Inc.[17]

V. APPLICATIONS OF VIBRATION-FREE CCR

Although originally introduced and still marketed primarily for Mössbauer spectroscopy, the effective vibration isolation capability of the DMX-20 interface makes it ideally suited to a wide variety of low-temperature applications such as micro-Raman, micro-PL, powder x-ray diffraction, RBS, and SIMS, in addition to a wide variety of scattering experiments using beams of neutrons or x rays. This is especially true of those techniques in which vibration causes a severe degradation in resolution. With appropriate modifications, the DMX-20 can be readily adapted to virtually any measurement technique and provide a reliable vibration-free platform for the most demanding measurements.

Any improvement due to reduced vibration levels may be marginal for macro-Raman applications. However, for micro-Raman and micro-PL measurements using a microscope, spatial resolution of 1 μm or more is routine. In such applications, the vibration-free feature of the DMX-20 interface is indispensable. Flow cryostats are routinely used in micro-Raman measurements largely because such cryostats are not only vibration-free but are easily moved to permit sample scanning, but naturally such systems require a supply of liquid helium. The DE202 CCR coupled to a cooled *XY* stage can open new possibilities of applications for micro-optical measurements. This is an area that is now receiving attention.

There are powder x-ray-diffraction configurations in which the x-ray beam and detector are moved simultaneously to record a scan while keeping the sample stationary either in a horizontal or in a vertical plane. Regardless of which geometry is used, the present DMX-20 interface can be adapted for use with such x-ray diffractometers. In such cases, the refrigerator-based cryostat may represent a somewhat higher initial investment than a traditional cryostat using liquid helium. But there can be little doubt that the reduced running cost of the CCR-based system will more than pay for itself in a relatively short time of a year or two of operation.

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APPENDIX

For a 25 μm thick natural Fe foil, taking density $\rho=7.86$ g/cm^3, the number of ^{57}Fe nuclei/cm^2 is found to be $n=4.55\times10^{18}$ cm^2. Taking the f factor (f) of α-Fe at room temperature of 0.83, the maximum resonant cross section for the 14.4 keV γ transition, $\sigma_0=2.57\times10^{-18}$ cm^2, the effective absorber thickness x for the inner two lines (possessing 1/12th of the total cross section σ_0) is found to be

$$x=n\sigma_0 f/12=0.80,$$

as shown by Visscher:[18]

$$\frac{\Gamma_{\text{obs}}}{\Gamma_s+\Gamma_a}=1+0.135x \quad \text{for } x\leq 5.$$

Since x increases in proportion to f upon cooling [see Fig. 4(b)] from 0.82 at 300 K to 0.92 at 10 K, one expects Γ_{obs} to increase.

Using $f(T=300\text{ K})=0.80$, we obtain $(\Gamma_s+\Gamma_a)=0.236/(1+0.135\times0.8)=0.213$ mm/s, which may be compared to $2\Gamma_n=0.196$ mm/s, the minimum observable linewidth based on Heisenberg's uncertainty principle.
At $T=10$ K, taking $f=0.92$, we obtain

$$\begin{aligned}\Gamma_{\text{obs}}&=(\Gamma_s+\Gamma_a)[1+0.135\times0.8\\&\quad\times f(10\ \text{K})/f(300\ \text{K})]=(0.213)(1.121)\\&=0.239\ \text{mm/s},\end{aligned}$$

which when compared to the Γ_{obs} at 300 K of 0.236 mm/s represents an increase of 0.003 mm/s. This is consistent with the observed linear increase of 0.004 mm/s in the temperature interval 10 K$<T<$300 K, yielding a slope 1.3×10^{-5} mm/s/K.

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