Thermal conductivity measurements of pitch-bonded graphites at millikelvin temperatures: finding a replacement for AGOT graphite

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Pitch bonded graphites are among the best known thermal insulators at sub-kelvin temperatures, but are very good conductors at higher temperatures. This makes them ideal for mechanical supports which must provide good thermal isolation at an operating temperature below 1 K, but must have good conductance at higher temperatures to aid in initially cooling down an instrument (a "passive heat switch"). One type of graphite, AGOT, has been known as having the lowest thermal conductivity below 1 K not only among graphites, but also compared with any other material. It is, however, no longer available. We have carried out thermal conductivity measurements at temperatures between 60 mK and 4 K on a proposed replacement, POCO AXM-5Q graphite, as well as a sample of AGOT graphite. Our measurements show that both graphites have a difference of about six orders of magnitude in conductivity between room temperature and 100 mK, but that AGOT graphite is not as good an insulator as previously believed. We conclude that AXM-5Q graphite is not only a suitable replacement for AGOT, but in fact is somewhat superior.

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1 Introduction

Pitch bonded graphites are among the best known thermal insulators at sub-kelvin temperatures. In contrast, they are very good conductors at higher temperatures, and at room temperature the thermal conductivity approaches that of pure metals such as copper and aluminium. This makes them ideal for mechanical supports which must be good thermal isolators at an operating temperature below 1 K, but must have good conductance at higher temperatures to aid in initially cooling down an instrument. Such components are sometimes referred to as passive heat switches.

Graphite is available in many different varieties, and the properties vary strongly depending on the source of the raw material and the manufacturing process, and thus on the ultimate composition [1]. The thermal conductivity of various pitch-bonded graphites[1] was measured at temperatures below 1 K in the 60's and 70's [4–6]. The lowest reported values were for AGOT graphite, a "nuclear" grade (i.e. designed for use in nuclear reactors) petroleum coke based high-purity extruded graphite [2]. Indeed, this material is believed to be one of the best known insulators at millikelvin temperatures. Materials which are better for reactor use are now available and it is no longer manufactured, though it is still occasionally used for its low temperature properties in labs which have retained supplies of the material [7]. A replacement would therefore be extremely useful.

One type of pitch-bonded graphite that has been well characterised at cryogenic temperatures is POCO AXM-5Q1. This is an industrial grade petroleum coke based fine-grained isotropic moulded graphite, graphitized at 2500°C [8]. Some typical properties are given in Table I. It was studied [8] in the 1980's with a view to using it as a standard reference material for thermal conductivity. This study included careful thermal conductivity measurements on two samples of the material over the temperature range from 5 K to room temperature. We have carried out measurements which extend the known temperature range to below 100 mK. However, we used a slightly different material, AXM-5Q, which is more readily available[2]. AXM-5Q1 differs only in that it has undergone an extra stage of purification to remove metallic impurities, and the electrical and thermal conductivity at room temperature are similar to AXM-5Q [8].

Mechanically, AXM-5Q graphite has superior properties to AGOT graphite, for example a compressive strength of 125 N mm^{-2} compared to under 50 N mm^{-2} for AGOT [9].

Despite the fact that slightly different conductivity values and different measurement temperature ranges have been reported in the millikelvin temperature range for AGOT graphite (for example in Ref. [10]), these all appear to refer to a single original measurement [4]. We therefore also measured the conductivity of a sample of AGOT from a small supply remaining in one of our laboratories in order to compare with the previous measurement.

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[1] Good descriptions of the manufacture and properties of pitch-bonded graphites are given in Refs. [2] and [3].

[2] Available from POCO graphite, 300 Old Greenwood Rd, Decatur, Texas 76234 USA

2 Experimental technique

The thermal conductivity of the two graphites was measured by the longitudinal steady heat flow method. A known power P was supplied to one end of the sample to establish a temperature difference $T_1 - T_0$ between the ends of the sample. By differentiation of the power (with T_0 constant during the measurement):

$$P(T_1) = \frac{A}{L}\int_{T_0}^{T_1} k(T)dT = g\int_{T_0}^{T_1} k(T)dT \qquad (1)$$

the thermal conductivity $k(T)$ can be obtained, where A and L are the cross-sectional area and length of the sample respectively, and g, the geometrical factor, is defined as $g = A/L$. Each data point was taken when stable values of both T_0 and T_1 were reached (this generally took about 30 minutes).

The experimental set up for the thermal conductivity measurement is shown in Fig. 1. The thermal contacts at the ends of the sample have been realised by means of two copper cylindrical blocks and two copper screws, 4 mm in diameter. Since the thermal contraction of graphite is lower than that of copper [11], the thermal contact between the blocks and the two ends of the sample becomes better on cooling. An SMD (Surface Mount Device) NiCr heater and a RuO_2 thermometer were glued onto the two copper blocks at the ends of the sample (see Fig. 1).

The electrical connections to the heater and to the thermometer were made with NbTi wires. The NbTi wires (25 μm diameter) were electrically connected by tiny crimped Cu tubes. At the ends of the NbTi wires a four lead connection was adopted. The bottom copper block was screwed onto a copper sample holder in thermal contact with the mixing chamber of a dilution refrigerator. Two RuO_2 calibrated thermometers were used for the measurement of T_1 and T_0. The thermometers were calibrated by means of an SRD 1000 (Superconductive Reference Device) and an NBS-SRM 767a fixed point device [12–14]. A copper shield, in thermal contact with the mixing chamber of the dilution refrigerator, surrounded the experiment. During measurements, the vacuum in the sample space was maintained at a pressure of 10^{-7} mbar or lower. The thermometers were measured using an AVS 47 a.c. resistance bridge and power was provided to the heater using a four wire $I - V$ source meter (Keithley 2601).

To ensure that the contact thermal resistances could be neglected, a second measurement run was carried out on both materials with a different geometrical factor g (about twice the original value). Within the experimental error, the same values of thermal conductivity were obtained in an overlapping temperature range.

Particle size	5 μm
Pore size	0.8 μm
Total porosity	23 %
Apparent density	1.73 g cm^{-3}
Compressive strength	125 N mm^{-2}
Tensile strength	50 N mm^{-2}

Table I: Typical properties for AXM-5Q graphite; data supplied by the manufacturer.

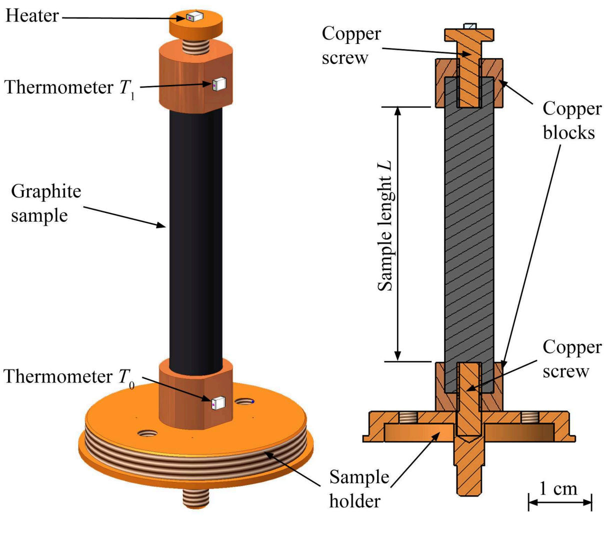

Figure 1: Set up for the measurements of the two types of graphite.

3 Results and discussion

3.1 Discussion

The sample of AXM-5Q graphite is a cylinder with a diameter of 11.04 ± 0.03 mm. The length (over which the temperature gradient is measured) is $L = 90.97 \pm 0.01$ mm, giving a room temperature geometrical factor of $g = A/L = 1.052 \pm 0.003$ mm.

The sample of AGOT graphite is a cylinder with diameter 8.00 ± 0.02 mm, length 42.01 ± 0.01 mm and a geometrical factor $g = 1.197 \pm 0.004$ mm. It was taken from a block marked with the lot number "XOT2K7". AGOT graphite exhibits some anisotropy[3] (there is a slight alignment of the crystallites with the layer planes parallel to the extrusion axis); the long axis of the cylinder (the direction in which the measurements were made) was parallel to the extrusion direction.

3.2 Details of calculating $k(T)$

For both samples, a plot of $P(T_1)$ was obtained (Eq. 1). The thermal conductivity was obtained by differentiation of $P(T_1)/g$. The measured thermal conductivity of AXM-5Q graphite in the 60 mK–3.25 K temperature range and of AGOT graphite in the 70 mK–4 K temperature range is shown in Fig. 3 2 and Table II.

There are three main contributions to the relative error in $k(T)$:

[3] This is one reason that AGOT is now considered to be obsolete [3].

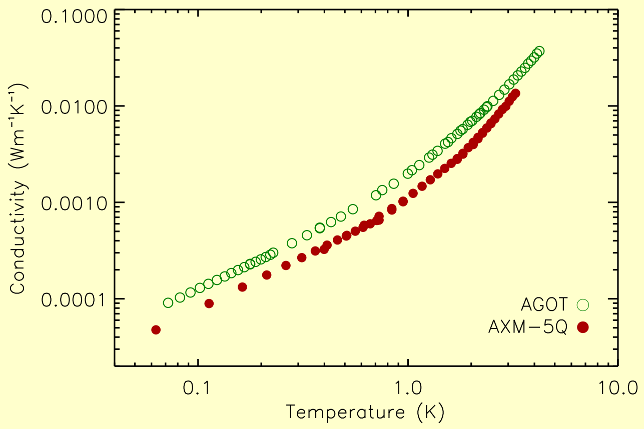

Figure 2: Measured conductivity of AGOT (○) and POCO AXM-5Q (●) graphite.

Temperature (K)	Conductivity ($Wm^{-1}K^{-1}$)	
	POCO AXM-5Q	AGOT
0.063	4.76×10^{-5}	-
0.072	5.51×10^{-5}	9.07×10^{-5}
0.080	6.18×10^{-5}	0.000101
0.090	7.02×10^{-5}	0.000114
0.100	7.85×10^{-5}	0.000127
0.120	9.54×10^{-5}	0.000153
0.140	0.000113	0.000179
0.160	0.000130	0.000205
0.180	0.000148	0.000233
0.200	0.000165	0.000258
0.250	0.000210	0.000333
0.300	0.000256	0.000408
0.350	0.000302	0.000489
0.400	0.000329	0.000578
0.450	0.000396	0.000660
0.500	0.000440	0.000755
0.600	0.000540	0.000964
0.700	0.000638	0.00118
0.800	0.000772	0.00144
0.900	0.000957	0.00169
1.00	0.00112	0.00199
1.10	0.00133	0.00233
1.20	0.00154	0.00269
1.30	0.00177	0.00309
1.40	0.00201	0.00351
1.60	0.00252	0.00456
1.80	0.00314	0.00564
2.00	0.00399	0.00693
2.25	0.00522	0.00873
2.50	0.00667	0.0109
2.75	0.00865	0.0134
3.00	0.0108	0.0163
3.25	0.0135	0.0197
3.50	-	0.0233
4.00	-	0.0321
4.22	-	0.0371

Table II: Measured conductivity of POCO AXM-5Q and AGOT graphite (the measured values are interpolated to a set of temperatures at fixed intervals).

- the power supplied to the sample: we estimate that the relative error of P is of the order of $\sim 0.1\%$;
- the measurement of the form factor g. The error in the measurements of g is estimated to be less than 1%;
- the uncertainty in the temperature due to the accuracy of the thermometers in this temperature range. A conservative value of $(\Delta T)/T$ is $\sim 2\%$ for $T > 1$K and $\sim 1\%$ for $T < 1$K.

Taking into account these contributions, the maximum relative error in $k(T)$ is about 3%.

We calculate that the total heat transfer due to conduction through the NbTi wires, convection by residual gas and thermal radiative exchange was of the order of 0.1% of the power supplied to the heater. These effects were therefore disregarded in the calculations, as was the correction due to thermal contraction ($\Delta g/g < 0.04\%$) [11].

3.3 Discussion

The measurements on AXM-5Q graphite are compared to other measurements in Fig. 3. Our results fall between limits obtained in a different laboratory on a specimen taken from the same rod [15] (the limits are approximately 40% higher and 25% lower than our measurements over the overlapping temperature range). Our results also appear to agree well with other measurements at higher temperatures [8], though this agreement should be taken with some caution for the following reasons. Firstly, the high temperature measurements were made on the purer material AXM-5Q1, rather than AXM-5Q as measured by us. In addition, measurements on AXM-5Q1 have shown considerable variation in thermal conductivity at the level of $\pm10\%$ between different samples of nominally the same material [8], and even as a function of position within a single sample [16]. We should therefore not expect better agreement than this between our measurements and other results. However, a similar problem usually occurs when comparing different thermal conductivity values for any material since measurements often suffer from systematic *errors* of this size.

Our measurements do not follow a simple power-law, and as temperature increases they appear to show a cross-over from a linear variation with temperature to a higher power-law exponent, consistent with the $T^{2.5}$ variation seen in other measurements at higher temperatures. (In general, exponents of 2.5 to 2.7 are seen for polycrystalline graphites [1]). We are not aware of other measurements showing the cross-over at lower temperatures for pitch-bonded graphites, although such behaviour is described in Ref. [6] without results being shown. However, this behaviour has been seen for highly oriented py-

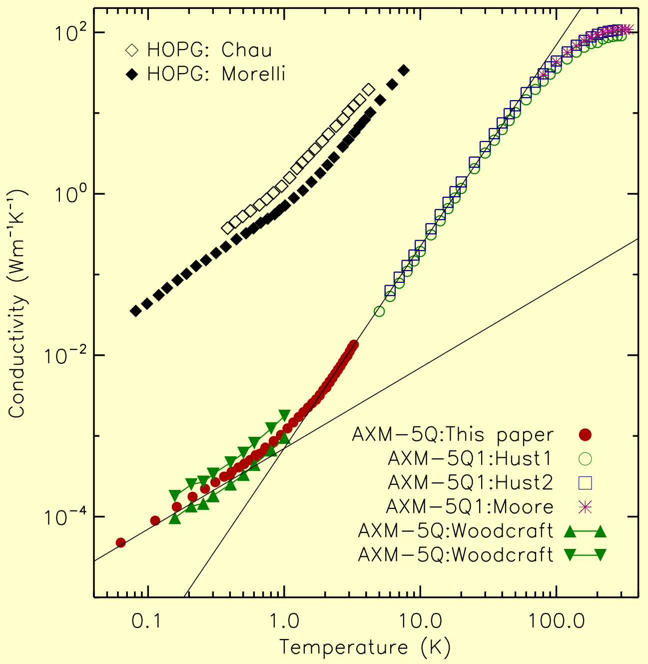

Figure 3: Measured conductivity of AXM-5Q graphite (•), along with upper and lower limits from previous measurements on a sample from the same rod [15] (▼, ▲), and measurements on three samples of AXM-5Q1 graphite [8] (○, □, ⋆). The solid lines show a linear and $T^{2.5}$ temperature dependence and are chosen to agree with the conductivity in the low and high temperature regimes respectively. Also shown: measurements on HOPG (highly oriented pyrolytic graphite) parallel to the layer planes [17] (◊), [18] (⧫)

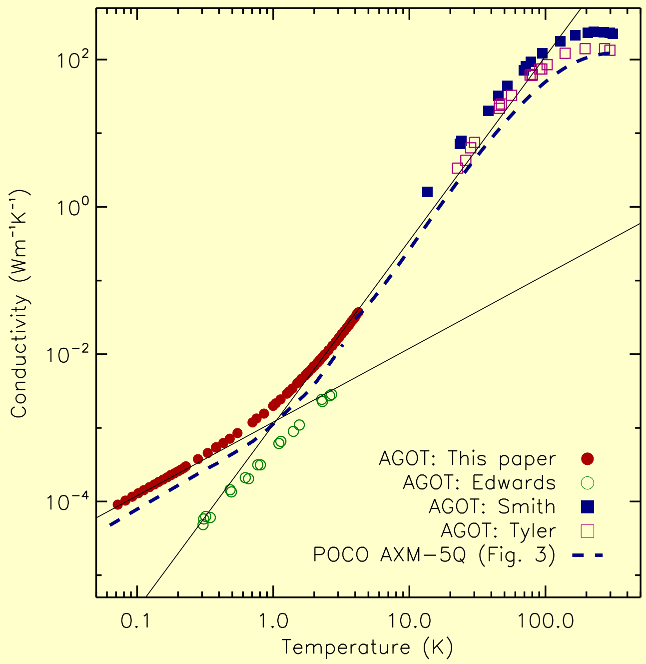

Figure 4: Measured conductivity of AGOT nuclear graphite parallel to the extrusion direction (•), along with other measurements from the literature: parallel to the extrusion direction below 10 K [4] (○) and above 10 K [19] (■), and perpendicular to the extrusion direction [20] (□). The solid lines show a linear and $T^{2.5}$ temperature dependence and are chosen to agree with the conductivity in the low and high temperature regimes respectively. Values for POCO AXM-5Q and -5Q1 graphite from Fig. 3 are also shown (dashed line).

rolytic graphite (HOPG).

The structure of graphite consists of planes of atoms (“layer planes” or “basal planes”) stacked together. Electrical conduction takes place within the planes, but not perpendicular to them. Thermal conduction in graphite is normally dominated by lattice (phonon) conduction. However, thermal conduction by electrons will take place whenever electrical conduction is possible. This is generally too small to be observed, but at sufficiently low temperatures the lattice thermal conduction falls sufficiently for electronic thermal conduction to dominate. All the graphites considered in this paper are polycrystalline. However, HOPG is made from crystallites which are well aligned with each other, so that the overall behaviour is similar to that of a single crystal. The electrical and thermal properties are thus highly anisotropic. Thermal conductivity measurements parallel to the layer planes [17, 18] show a near linear temperature variation of conductivity below 1 K, corresponding to electronic thermal conduction (Fig. 3). Pitch-bonded graphites are also polycrystalline, but the crystal axes are not highly oriented, and electrical conduction is therefore possible in all directions, taking place via crystallites that are favourably oriented. We would therefore expect the behaviour of pitch-bonded graphites to be similar to that of HOPG in-plane, as we observed.

We see similar behaviour for our measurements on AGOT graphite (Fig. 4), and an extrapolation of the conductivity to higher temperatures using the expected $T^{2.5}$ behaviour is in reasonable agreement with results from the literature. As with AXM-5Q graphite, we would not expect perfect agreement due to systematic experimental errors and sample to sample variations. Both our measurements and those at higher temperature show that AGOT has a slightly higher conductivity than AXM-5Q graphite.

However, the conductivity values previously reported below 1 K [4] are much lower. Furthermore, they show a different temperature dependence and do not seem consistent with the higher temperature data. It appears that the results from Ref. [4] are based on a single set of measurements, in which the effect of contact conductance was not determined. A possible explanation is that the results suffer from a systematic error. Thermal contact was made using grease, and a significant thermal resistance at the contacts would cause the overall measured conductance to drop, and to show a greater than linear temperature dependence as observed.

However, the measurements shown in Fig. 4 were all made on different samples, and we have no knowledge of sample to sample variations. Indeed, there are different types of AGOT graphite [2]. The sample measured in Ref. [19] was

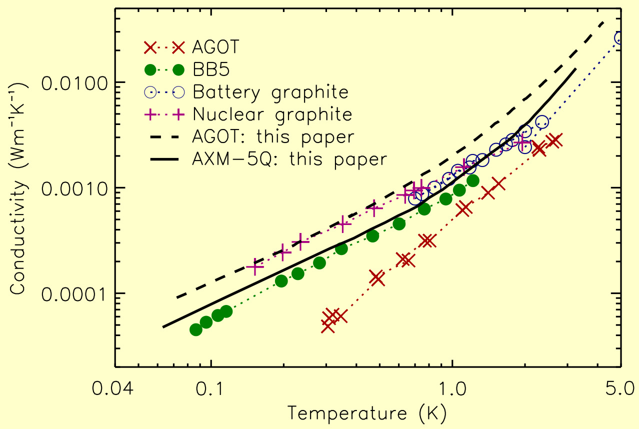

Figure 5: Comparison of the conductivity of graphites measured by us and from the literature. Our measurements: AGOT (dashed line), POCO AXM-5Q (solid line). Other measurements: AGOT [4] (×), BB5 [5] (•), "battery graphite" [6] (○), CEN nuclear graphite [5] (+). The points for each measurement are joined by dashed lines for clarity.

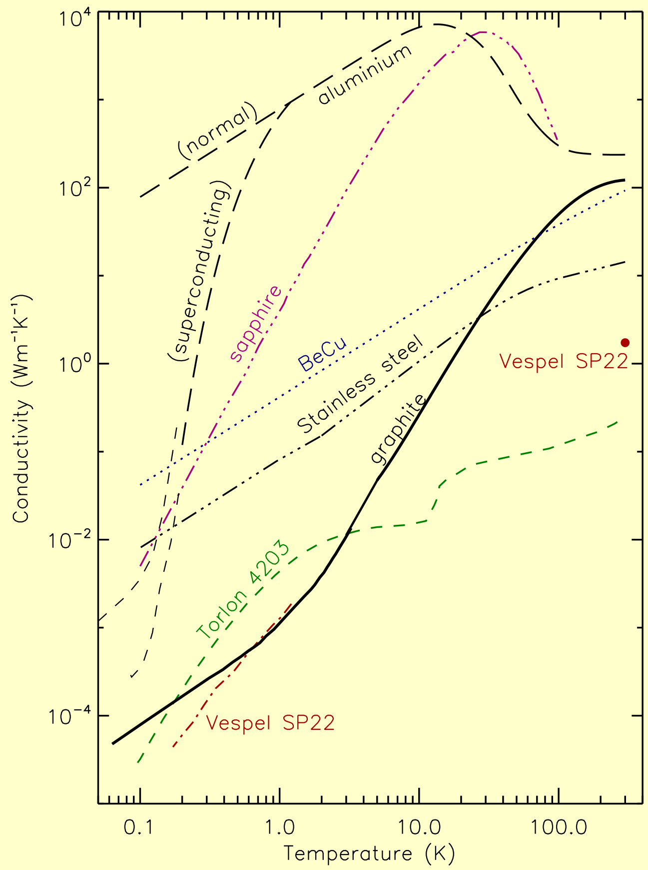

Figure 6: Comparison of the conductivity of AXM-5Q and 5Q1 graphite with representative values for other materials: aluminium [21], sapphire [22] (extrapolated below 400 mK), high strength beryllium copper (C17200) [23], 18-8 stainless steel [24, 25] (interpolated between 1 and 2 K), Vespel® SP22 [5, 26] and Torlon® [27, 28]. Nylon is another good insulator at cryogenic temperatures; the conductivity is similar to Torlon®. For Vespel® we are not aware of measurements between 1 K and room temperature; a single value for the room temperature conductivity is shown (•), linked by a straight line to the low temperature values. The aluminium values are taken from Ref. [21], and (in the normal state) apply to an RRR of 850, which is the highest value expected for 4N (99.99%) purity [21]. In the superconducting state, variations in lattice conductivity between samples are large [21], and do not appear to correlate with sample purity. The two sets of values shown correspond to the two extremes seen in Ref. [21].

described as the variant AGOT-KC, but the remaining measurements, along with the shipping note accompanying our sample, merely refer to AGOT.

We therefore cannot rule out sample to sample variation as an explanation for the discrepancy. Unfortunately the two measurements above 10 K shown in Fig. 4 are not directly comparable, since AGOT graphite exhibits considerable anisotropy and they were taken perpendicular to each other with respect to the extrusion axis (a similar ratio of conductance between the two measurement directions has been seen for room temperature measurements on AGOT [2]). However, our measurements and those of Ref. [4] were both made parallel to the extrusion axis, and should not differ for this reason.

A comparison with measurements from the literature on other graphites below 1 K (AGOT [4], BB5 [5], "battery graphite" [6] and CEN nuclear graphite [5]) is shown in Fig. 5. With the exception of the AGOT results, discussed above, they show a similar magnitude and temperature dependence of conductivity to our measurements, suggesting that the behaviour we have observed is quite general. It is almost certain that all the graphites shown here are pitch-bonded, though we only know this to be the case for the AXM-5Q, AGOT and battery graphite measurements.

3.4 Comparison with other materials

The thermal conductivity of graphite is quite unusual; to put this in context, Figure 6 shows how the conductivity of other materials compares with graphite. The values for aluminium, beryllium copper (BeCu) and stainless steel demonstrate the behaviour of metals. The purest metals show a large conductivity peak below room temperature, resulting in conductivities at millikelvin temperatures that can be similar to room temperature. The ratio of room temperature to millikelvin conductivities, referred to in the following discussion as the "conductivity ratio," is therefore small. As the purity decreases, the peak vanishes, and materials such as beryllium copper have a nearly linear temperature dependence. Lower conductivity (less pure) alloys such as stainless steels have even smaller conductivity ratios.

An exception is metals which exhibit superconductivity, and a corresponding sharp drop in thermal conductivity below the superconducting transition temperature. For example, aluminium has a similar difference between conductivity at 100 mK and room temperature as AXM-5Q graphite. However, since the aluminium conductivity is still very high at 1 K, it would only be useful in isolating two parts of an instrument which were both at millikelvin temperatures; such a situation is rarely encountered. The problem with aluminium arises because the superconducting transition temperature is so low. Two commonly encountered elements, lead and niobium, have higher transition temperatures. However, the peak in thermal conductivity occurs near to their transition temperatures, and consequently the difference in conductivity between room temperature and 1 K is only about one order of magnitude [29], making them no more useful than aluminium. These materials do find a use when used as an active heat switch, with an applied magnetic field used to switch them from the superconducting (insulating) to normal (conductive) state. Such switches suffer from the problem of relatively low conductivity at the higher temperatures encountered while an experiment is cooling down, which can be alleviated by putting graphite in parallel [30].

Crystalline dielectrics such as sapphire show a conductivity peak similar to pure metals; polycrystalline materials such as alumina have much lower conductivities but still show a similar peak. Insulators such as Vespel® SP22, Torlon® 4203 and nylon show similar conductivities to AXM-5Q graphite below 1 K, but do not reach such high conductivities at room temperature.

Pitch-bonded graphites are therefore unusual in providing such a large conductivity ratio (six orders of magnitude from 300 K to 100 mK). The reason for this is that the thermal conductivity peak occurs near room temperature, giving the largest possible conductivity ratio. For graphites with higher conductivities, the peak occurs at lower temperatures, reducing the conductivity ratio.

Graphites with smaller crystallites have even lower conductivities than AXM-5Q graphite [19, 20], presumably because the phonon mean-free-path is limited by scattering from the crystal boundaries. However, electron mean-free-paths are much smaller than for phonons, and the reduction in electronic thermal conduction is likely to be less than in lattice conduction, reducing the conductivity ratio. This is hinted at by the results in Ref. [6] for battery graphite. The conductivity below 1 K is similar to AXM-5Q graphite, while the room temperature value can be estimated from the quoted electrical resistivity. Various relationships have been proposed between thermal and electrical conductivity at room temperature [2, 31], and for this material they suggest a conductivity of around 16 $\mathrm{Wm^{-1}K^{-1}}$ or lower, which is considerably lower than for AXM-5Q graphite. This suggests that AXM-5Q has a nearly optimal conductivity ratio.

4 Conclusion

Pitch-bonded graphites are good thermal insulators at low temperatures but good conductors near room temperature. One type of graphite, AGOT, has been known as the best insulator below 1 K not only among graphites, but also compared with any other material. However, it is an obsolete material and is no longer produced. We measured the conductivity of a possible replacement, POCO AXM-5Q graphite, as well as of a sample of AGOT graphite remaining in one of our labs.

Our results were generally consistent with other measurements from the literature. The measured conductivities of the two samples were similar to each other and also to measurements on other pitch-bonded graphites in the literature. Both showed a cross-over from a linear temperature dependence below 1 K, believed to be due to electronic thermal conductivity, to a higher power-law at higher temperatures where lattice conduction is presumed to dominate. Similar behaviour has been seen in other types of graphite with much higher conductivity. Measurements on both the samples were in good agreement with values from the literature at higher temperatures. In both cases the conductivity falls by approximately six orders of magnitude from room temperature to 100 mK, making them highly suitable for supporting millikelvin stages in instruments.

The one major discrepancy is that the conductivity previously reported [4] on AGOT graphite below 2 K is somewhat lower than our measurements, and shows a different temperature dependence. This could be due to variations between our samples, but we suspect that the original measurements were in error due to the measurements including a non-negligible contact resistance. If this is the case, then AGOT graphite is not in fact the best known insulator at millikelvin temperatures; Vespel® SP22 is a better insulator below approximately 600 mK. However, this behaviour is only achieved by the addition of graphite; *pure* Vespel® has a significantly higher conductivity [5].

Based on our measurements, AXM-5Q graphite has similar (and indeed slightly better) insulating properties to AGOT. More importantly, it has superior mechanical properties. We therefore consider AXM-5Q graphite to be not only a suitable replacement for AGOT graphite, but a considerable improvement.

Acknowledgement

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