PRELIMINARY TESTS ON SUPERCONDUCTIVITY II

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1. **Introduction.**

In a previous note \(^1\) we have described some tests on superconducting Nb-Zr alloys in wire form performed with the aim of gaining some experience in view of the possible use of superconductors for the construction of electromagnets.

We have continued our investigation, trying to enlarge somewhat the field of the previous approach, by studying new materials and techniques: we report here the results, together with a description of the low temperature equipment power supplies and control circuits which have performed satisfactorily.

In Appendix III we give a detailed account of the behaviour of Hall plates at liquid helium temperature.

2. **Experimental equipment**

2.1 **Cryostat**

Two cryostats for liquid helium have been designed and successfully tested.

One consists of two double walled evacuated coaxial containers made out of argon ARC welded stainless steel components, (Fig. 1). Adsorbent charcoal is included in the vacuum chamber of the inner dewar. Evacuation valves of the Richards-type \(^2\) are used to seal both vacuum chambers.

The second is a narrow tail cryostat to be used in connection with a magnet, (Fig. 2). It consists of cylindrical elements made out of polished stainless steel or copper. A copper radiation shield kept approximately at liquid nitrogen temperature by heat conduction, surrounds the narrow tail. An adsorbent pocket is fitted in the evacuated volume, which is sealed off by a 10 mm dia. Leybolt valve.

Both dewars can be connected to a system of pipes for exhausting pumping and transferring liquid helium. The transfer line discharges liquid helium right to the bottom of the cryostat to allow for more efficient precooling of the samples by helium cold vapour. The line is also used to support the samples and the liquid level indicating resistors, (Fig. 1).

2.2 **Transfer line**

The transfer line, designed for use in connection with the cryostats of the preceding section, consists of two separate parts. One part is fixed to the cryostat
assembly. It consists of a stainless steel tube, 7 mm i.d., 8 mm o.d., vacuum
ejacketed down to 0.3 m in the cryostat's neck. It can be extended to the bottom
of the cryostat by the addition of a removable stainless steel pipe.

The second part is mounted on the storage dewar and consists of vacuum
jacketed stainless steel pipe 7 mm i.d., 8 mm o.d. Dipping tubes of appropriate
length can be mounted to extend the line down to the bottom of storage containers
of different sizes. The flow of helium can be regulated or interrupted by adjusting
a valve operated by turning a screw placed at the neck of the container. The
vertical part of the line above the neck of the dewar, together with the horizontal
part, has a radiation shielding at liquid nitrogen temperature, obtained by cooling
the outer wall of the vacuum jacket which is in good thermal contact with a
parallel pipe containing liquid nitrogen. Parts of liquid nitrogen temperature are
insulated in styrofoam shells.

Both elements of the line are evacuated and sealed off with Richards-type
valves. The continuity of the two parts is established by a Johnston-type joint.

Fig. 3 shows some of the characteristics of the line.

2.3 D.C. magnet

A water cooled D.C. magnet has been designed to produce an axial magnetic
field in the narrow tail of the cryostat of section 2.1. The magnet consists of
14 layers copper band 10 x 3 mm², each layer having 10 and 1/4 turns. The spacing
between adjacent layers is 1 mm to allow axial flow of cooling water: the nylon
spacers consist of 5 x 1 mm² bars having on one of the wider faces appendices 1 mm
thick, 3 mm long, which are inserted between windings for electrical insulation.
A pressure switch and a thermostat mounted at the water inlet and outlet respectively,
are connected to the controls of the generator.

The field in the centre of the magnet has been measured by means of a
Hall plate Siemens PC 33: its value is 1.03 ± 1.5 c/o Wb/m² per 1000 A. The
maximum current which has been sent through the magnet has been limited to 3.150 A
by the generator available. At this current, corresponding to a field of 3.2 Wb/m²
the overall increase of the water temperature was about 10 degrees.

Fig. 4 shows an axial section of the magnet with the narrow tail cryostat
in position.

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2.4 Power supply

2.4.1 We have modified the power supply described in reference 1) in order to obtain two different modes of operation, i.e., as a current or voltage generator. In both cases the power supply can deliver a continuous current up to 100 A, or square current pulses of 200 A with maximum pulse duration of 0.5 s and duty cycle 1/2.

Fig. 5 shows a circuit diagram for the former case. The main source of power is an 8 V storage battery, 400 Ah. A 24 V battery tapped at 6 V provides the auxiliary current for the control circuits. Transistors T1 and T2 form a differential amplifier. An adjustable reference voltage is applied to the base of T1, and the voltage drop across resistor R, in the emitter circuit of the output stage, is applied to the base of T2. This voltage is approximately proportional to the current in the load, which is connected in series with the collectors of the output stage, T7 to T14.

For each setting of the reference voltage, the differential amplifier tends to maintain the voltage drop across R at a certain value, and hence the current in the load, through the action of the cascaded current amplifier stages T3, T4, T5 - 6 and T7 - 14. The current in the load can be varied by changing the value of the reference voltage by means of P1.

Such a power supply can be regarded as a current generator, because the dynamic output impedance is of the order of 500 ohms, i.e., much in excess of the load impedance. Therefore the current which flows through the sample is practically independent of the variations of resistance of the connecting leads provided that the voltage drop across the load does not exceed 4 volts. Furthermore, the different heating of the connecting leads, occurring when duration or repetition of the current pulses are changed during measurements, does not substantially influence the preset value of the current.
A drawback of this arrangement lies in the fact that, if the sample carrying a current $I$ becomes normal, presenting a low resistance $R_{\text{norm}}$, such that $R_{\text{norm}} \cdot I$ is smaller than $4 \, \text{V}$, the current does not decrease, and the dissipation $R_{\text{norm}} \cdot I^2$ leads rapidly to the destruction of the sample.

2.4.2 To prevent this danger, the power supply has been modified to achieve an instantaneous reduction of the current to a negligible value in case of a marked increase in the resistance of the sample. Fig. 6 shows the modified parts of the power supply. The load is now placed in the emitter circuit of the output stage, in series with the resistor $R$.

Due to the feedback action through $R_f$, the differential amplifier stage T1 - T2, manages to keep the voltage drop across $R_c$ constant, i.e. the current in the load. But as soon as the load is no longer superconductive, the voltage drop across it assumes a value many times greater than the voltage drop across the connecting leads. At this moment the low voltage Zener diode $Z$ becomes conductive, and the voltage build up across the load is applied to the base of T2. This has the effect of lowering the output current to practically zero, i.e. the leakage current of T7 to T14.

By this means, one combines the advantage of a constant current source with an automatic undelayed protection against undue dissipation in the sample.

2.4.3 Transistor T15 has been added to allow automatic pulse operation: it acts as switching stage, (see fig. 5).

When T15 is biased to full conduction, point "A" is clamped at ground potential, and therefore T3 and the following stages are cut-off: no current flows in the load.

If a positive square pulse is applied to the base of T15, the transistor is cut-off, and, during a time equal to the pulse length, point "A" becomes free to follow the output signal of the amplifier T1 - T2: the current in the load will assume the value preset by adjusting P1.
A NAGARD type 5002 was used as a pulse generator. Pulses of any length above 0.5 ms could be obtained in single shot or repetitive operation with the limitations reported in section 2.4.1.

A power diode was connected across the sample to absorb the voltage transient, which is generated when switching off the current if the load is inductive. If the current is switched off while the sample is in the superconducting state, the energy stored in the magnetic field will be dissipated mostly in the diode; but if the switching of the current occurs because superconductivity is quenched in the sample most of the energy will be dissipated in the sample itself.

The dissipation in the sample can be reduced by inserting a resistor in series with the diode; however, in this case, overvoltage and breakdown might occur.

2.5 Nitrogen level regulation

2.5.1 An automatic filling system was designed to control the liquid nitrogen level in the radiation shield of the cryostats. The details of the electric circuit are illustrated in Fig. 7.

The sensitive elements are two 1/4 watt carbon resistors R1 and R2 from which the protective resin varnish has been removed. Their room temperature resistance is $470 \pm 10$ ohm, rising to about 700 ohm when immersed in liquid nitrogen.

These two resistors are placed inside of the nitrogen dewar close to the top of the inner vacuum chamber, (see Fig. 1), vertically spaced by a few centimetres. The resistance of R1 and R2, mounted as described, and completely immersed in liquid nitrogen, differs by about 10 ohm from the resistance they assume when they are just above the liquid nitrogen level.

2.5.2 When the upper resistor is immersed, the nitrogen level is correct. In this condition the bridge composed of R1, R2 and P1 is balanced to give zero d.c. voltage at the input of the amplifier stage. (See Appendix I). The amplifier stage is necessary in order to keep the current flowing in R1 and R2 very low (about 4 mA)

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to avoid excessive Joule heating. Under these circumstances the output of the amplifier is zero, and the polarised relay $K_1$ is in its middle position.

When the evaporation of the liquid nitrogen has lowered the level and the upper resistor is out of the liquid, relay $K_1$ becomes energised and closes its upper contact.

The auxiliary relay $K_2$ is also energised and opens the filling valve $K_3$, which connects a liquid nitrogen container to the cryostat: the level in the dewar is restored, and when the normal level is reached relays $K_1$ and $K_2$ are de-energised, and the valve $K_3$ closes again.

A meter connected to the output of the amplifier gives an approximate indication of the level.

Switch $SW_1$ permits the switching off of the automatic circuit and the manual operation of the valve $K_3$.

2.5.3 Liquid helium level monitor: the position of the liquid helium level was monitored by means of 3 resistors mounted in the inner dewar, at three different heights.

The 3 resistors were fixed to the transfer line, which feeds liquid helium to the bottom of the dewar, (see Fig. 1).

Standard $1/4$ watt carbon resistors have been used, after removing of the resin encapsulation. Their room temperature resistance of $510 \, \Omega$ nominal, rises to about $26,500 \, \Omega$ at liquid helium temperature. The resistance falls by about $8 \%$ when lifting an immersed resistor just above the liquid level.

3. Experimental results

3.1 Test of samples of wire in the absence of an external magnetic field

Tests of samples of wire were performed bearing in mind the following objectives:

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a) to obtain a practical means of connecting Nb-Zr wires to the copper power leads, different from ultrasonic soldering with indium.

b) to find a method of connecting two superconductors in a superconductive joint.

c) to test the properties of new materials.

Ultrasonic soldering with the addition of indium has proved to be a successful method to join superconductors to copper leads and a substantial current can be achieved in a joint provided that a sufficient surface is foreseen to dissipate the heat developed in the joint. For all practical applications of superconducting wires in winding coils, the high magnetic field will considerably reduce the maximum current carried in the superconducting state in comparison with the current that could be maintained in a weak magnetic field: thus the requirements on the connection to the power leads become less stringent and a less efficient method than ultrasonic soldering with indium may be envisaged, provided that it is simple and reproducible. We have tested a junction obtained by introducing the copper (0.5 mm diameter) and the niobium (0.25 mm diameter) wires in a nickel tube (0.5 mm i.d.; 0.8 mm o.d.) and by welding the copper and niobium to the nickel tube by passing an electrical current perpendicular to the axis of the tube at a few points.

The method has proved to be successful up to currents of 22.5 A. The addition of an indium cylinder around the joint considerably increased the current carried.

Spot welding with a flame in a reducing atmosphere has proved to be a simple means of joining two superconducting wires even of different characteristics as Nb with Nb - 25 o/o Zr, with a reasonable uniformity of the results at least within the limited statistics of our investigation.

An alternative method of joining two superconductors has been the use of ultrasonic soldering of the wires with an alloy of Pb + 5 o/o In. The method has proved to be particularly satisfactory with Mo-Re wires allowing a current of 175 A in the joint; with Nb and Nb alloys currents up to 28 A have been passed through. However, such an approach is possibly inadequate when a magnetic field is applied to the joint.

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Of the new materials tested an 0.1 mm Nb-25 o/o Zr wire produced by METALLGESELLSCHAFT, A.G., Frankfurt – Main carried a maximum current of 58 A; yet nothing can be said about its uniformity since the three samples tested had remarkably different performances.

A second interesting material tested was a Mo-50 o/o Re 0.3 mm wire produced by BOCUZE ET CIE, Lyon. In the absence of an external magnetic field, the wire carried currents up to 175 A, this value probably being determined by heat generation in the connections to the power leads.

In Appendix II additional information concerning the tests described in this section is reported, together with a table giving the characteristics of all the samples.

3.2 Tests of samples of wire in a longitudinal magnetic field

Samples of superconducting wire were mounted parallel to the axis of the magnet described in section 2.3. The samples were indium soldered to copper power leads 0.5 mm in diameter with the addition of indium cylinders 25 mm long, 4 mm ⌀ around the joints. Voltage leads were soldered about 2 mm away from the indium cylinders, to measure a voltage drop along 20 mm of superconducting wire. The transfer tube supported the sample by means of the clamps fixing the indium joints, which in turn kept the superconductor in place. The samples were located with a precision better than ± 1 mm both axially and radially; taking into account the field distribution in the centre of the magnet, this means that the field acting on the wire had a uniformity better than 1 o/o.

Current density vs. magnetic field curves for the three samples are shown in Fig. 8.

3.3 Superconducting coil

The coil consisted of 1300 turns of 0.254 mm Nb-25 o/o Zr wire produced by WAH-CHANG CORP., Albany, Oregon. This wire was insulated with a double silk lapping which increased the wire diameter to about 0.33 mm. The wire was wound on a bakelite former determining the coil’s inner diameter and axial length of 10 and 15 mm respectively. The outer diameter turned out to be 28 mm, with a total wire length of approximately 78 m.

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Electrical connections of the power leads to the coil terminals were obtained by indium ultrasonic soldering with the further addition of indium around the joint, in the form of cylinders 40 mm long, 7 mm in diameter.

The maximum current that could be passed through the coil in the superconducting state was 24.5 A i.e. a current density of 482 A/mm².

The value of the magnetic field in the centre of the coil was measured with a Hall plate Siemens SBV 524: at a current of 24.5 A the field was 1.74 Wb/m² ± 1.5 o/o.

3.4. Persistent current circuit

The circuit consisted of a coil of 150 turns of 0.254 mm Wah-Chang Nb-25 c/o Zr wire wound on a stainless steel former 10 mm o.d., 12 mm long. The coil's terminals were ultrasonically indium soldered to the power leads with the addition of indium around the joints.

Niobium wire 0.127 mm in diameter was used to short-circuit the terminals. This wire was soldered to the Nb-Zr wire of the coil using a Pb-5 c/o In alloy and was located in the gap of an independently powered C magnet.

Persistent current could be established in the circuit either by powering the C magnet, sending current through the coil, disconnecting the C magnet and disconnecting the power supply of the coil; or by sending current in the Nb wire, powering the C magnet to switch the current to the coil, disconnecting the C magnet and the power supply of the coil respectively.

The existence of a persistent current was checked by measuring the magnetic field of the coil with a Hall plate Siemens FC 33.

Currents up to 27 A could be maintained for times up to 2 h 40, the time limitation being established by liquid helium losses mostly in the electrical copper leads.

/PV

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The d.c. amplifier described in Appendix I has been developed and tested by Mr. M. Grütter.

We acknowledge also the work of Mr. G. Toninato in the construction of the cryostats and of Messrs. P. Actis, M. Grossi and H. Lenique in the construction and tests of our instrumentation and samples.

REFERENCES

1) P.G. Innocenti and G. Kuhn - Preliminary tests on superconductivity NPA/Int. 62-1, 16th January 1962

APPENDIX I

A TRANSISTORIZED D.C. AMPLIFIER

The d.c. amplifier consists of a differential stage $T_1 - T_2$, followed by a twin emitter follower stage, $T_3 - T_4$ (see fig. 9).

$T_1$ and $T_2$ will usually have different characteristics. To obtain zero output for zero input two independent controls are foreseen : $P_1$ for the differences between base-emitter voltages, and $P_2$ for the differences between current amplification factors.

The drifts due to ambient temperature changes are minimised by mounting the two transistors close together in a metal block, and operating the stage at a low working current, accepting some loss in the resultant current amplification factor.

$P_3$ is used to adjust the gain at the desired value.

The amplifier power supply is not grounded, so that, in the case of a floating load, either one of the terminals, or an intermediate point of the source can be grounded, without affecting the operation of the amplifier.

The linearity is better than 1 o/o for output currents up to 100 µA.

For $20^\circ$ C temperature rise above room temperature the drift referred to the input was about 0.1 µA, with open circuit input, and negligible with short-circuit input.
APPENDIX II

DETAILED PRESENTATION OF RESULTS OF TESTS OF SEC. 3.1

For the interpretations of the results listed in table I it is helpful to put forward some observations about two possibly different types of transition from the superconducting to the normal state when square current pulses are sent to a wire sample. We call a sharp (S) transition one which occurs in a fraction of a millisecond, that is in a time of the same order of the rise time of the current pulse: it might imply a quenching due to excessive current density. A delayed (D) transition on the other hand is one which occurs some milliseconds after the current has reached its highest value: it might be explained by excessive Joule heating in the joints between superconducting wires and power leads which increases the temperature of the superconductor near the transition temperature where current densities allowed are lower.

We have used this criterion in column 6 of table I.
APPENDIX III

BEHAVIOUR OF HALL PLATES AT LOW TEMPERATURE

We have measured the characteristics of three Siemens Hall plates at liquid helium temperature and we have plotted in Fig. 10 and Fig. 11 the Hall voltage as a function of the magnetic field for a control current of 100 mA. For comparison, room temperature characteristics, and in one case liquid nitrogen temperature characteristics, have been plotted on the same figures. The Hall voltage having been measured by comparison with a known voltage with zero current flowing between the Hall terminals of the plate, the temperature dependence obtained is independent of the temperature variation of the resistance between the Hall terminals. The precision of the measurements is limited by the uncertainty of ±1.5 o/o in the value of the magnetic field in the centre of the magnet.

The Hall voltage versus magnetic field curves for plate SBV 524 and plates FC 33 have a different temperature dependence. For the former plate the curve steepens in passing from 4.2° K to 77.3° K and to ambient temperature, for the latter two plates the curve is less steep at ambient temperature than at liquid helium temperature.
from pulse generator
Z out = 100Ω

SW1
1. STOP
2. PULSED CURRENT
3. CONTINUOUS CURRENT

NOTE:
The 8 V - storage battery which delivers the main current, is not grounded.

CONTROLLED CURRENT GENERATOR

Fig. 5
CONTROLLED CURRENT GENERATOR

MODIFIED PARTS WITH AUTOMATIC PROTECTION CIRCUIT ADDED

Fig. 6

16.5.62 Mattu
R1 – R2: 1/4 W carbon resistors, resin cover removed
nominal 470Ω, about 700Ω in liquid N₂

AUTOMATIC N₂ FILLING SYSTEM

Fig 7
- All transistors type BCZ 11
- T1 and T2 selected for equal and maximum $\beta$ at $I_C = 70 \mu A$
- and $V_{CE} = 3V$
- P1 = open circuit balance
- P2 = short circuit balance
- P3 = gain adjust.

- $Z_{input} = 10 \, k\Omega$
- $Z_{output} = 100 \, \Omega$
- Voltage gain $50$

**D.C. Amplifier**

Fig. 9
HALL PLATES SIEMENS FC 33
No 634 and No 630
CONTROL CURRENT 100 mA

Fig. 11
<table>
<thead>
<tr>
<th>Sample description</th>
<th>Joint between superconductor and copper leads</th>
<th>Indium added at the joint ( \leq 7 ) mm 40 mm long</th>
<th>Max current (A)</th>
<th>Max time at max currents (sec)</th>
<th>Transition current and type</th>
<th>Remarks</th>
</tr>
</thead>
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<tr>
<td>1 Wah-Chang Nb 25%, Zr wire ( \phi 0.2 ) mm</td>
<td>Indium ultrasonic soldering</td>
<td>yes</td>
<td>200</td>
<td>0.10</td>
<td>200 D</td>
<td>For a longer time the wire melts.</td>
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<td>2 id</td>
<td>Nickel tube, electrical point welding</td>
<td>no</td>
<td>50</td>
<td>1</td>
<td>55 D</td>
<td>Transition after 10 sec</td>
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<tr>
<td>3 id</td>
<td>id</td>
<td>no</td>
<td>22.5</td>
<td>1</td>
<td>27.5 D</td>
<td>Transition after 10 sec</td>
</tr>
<tr>
<td>4 id</td>
<td>id</td>
<td>yes</td>
<td>100</td>
<td>1</td>
<td>105 D</td>
<td>Transition after 2 sec</td>
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<td>5 id</td>
<td>id</td>
<td>yes</td>
<td>100</td>
<td>1</td>
<td>102 D</td>
<td>Transition after 3 sec</td>
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<td>6 id</td>
<td>id</td>
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<td>140</td>
<td>1</td>
<td>145 D</td>
<td>Transition after 2 sec</td>
</tr>
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<td>Indium ultrasonic soldering</td>
<td>yes</td>
<td>23</td>
<td>1</td>
<td>25 D</td>
<td>Transition after 18 sec</td>
</tr>
<tr>
<td>8 Metallgesellschaft Nb 25%, Zr, ribbon ( \phi 0.15 \times 0.3 ) mm</td>
<td>id</td>
<td>yes</td>
<td>100</td>
<td>0.1</td>
<td>102 D</td>
<td>Transition after 20 sec</td>
</tr>
<tr>
<td>9 Metallgesellschaft Nb 25%, Zr, wire ( \phi 0.1 ) mm</td>
<td>id</td>
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<td>58</td>
<td>0.02</td>
<td></td>
<td>After quenching twice at 600 the wire burnt</td>
</tr>
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<td>10 id</td>
<td>id</td>
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<td>55</td>
<td>0.2</td>
<td>( &gt; 55 ) S</td>
<td>After two quenching the wire burnt</td>
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<tr>
<td>11 id</td>
<td>id</td>
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<td>10</td>
<td>1</td>
<td>( &gt; 10 ) S</td>
<td></td>
</tr>
<tr>
<td>12 Bocuse Mo, 50% Re, ( \phi 0.3 ) mm</td>
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<td>152</td>
<td>1</td>
<td>165 D</td>
<td>Transition after 6 sec</td>
</tr>
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<td>13 Flame welding between two Wah-Chang Nb 25%, Zr wires ( \phi 0.254 ) mm</td>
<td>id</td>
<td>yes</td>
<td>72.5</td>
<td>1</td>
<td>75 S</td>
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<td>S</td>
<td>I</td>
<td>26</td>
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</table>

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**Notes:**
- **Temperature:**
  - 100°C max
  - 60°C min
- **Current:**
  - 25 A max
  - 5 A min
- **Voltage:**
  - 60 V max
- **Duration:**
  - 1000 ms

**Sample Description:**
- Superseded by
- Updated by
- Modified by
- Reviewed by
- Revised by

**Revision History:**
- 0.15 x 0.3 mm
- 0.1 x 0.2 mm
- 0.05 x 0.1 mm

**Notes:**
- The table is for reference only.
- The values provided are approximate.