A.5: Development of residual resistivity ratio measurement facility for niobium

Advanced Accelerator Module Development Division, along with Magnetic and Superconducting Materials Section of RRCAT, has developed a Residual Resistivity Ratio (RRR) measurement facility aimed for upcoming project of development of Super-Conducting Radio-Frequency (SCRF) technology for accelerator applications. Majority of the SCRF cavities are made out of bulk niobium (Nb) of a defined purity usually specified by its RRR as one of the important parameter for achieving desired accelerating gradients. The measurement of RRR is one of the popular and most convenient techniques for gross qualification of Nb.

A stable performance of a SCRF cavity requires materials having high thermal conductivity so that accidental heat generated at local hot spots is carried away quickly to the liquid Helium (LHe) bath. As postulated by the Wiedemann-Franz law, the thermal conductivity of a metal at a particular temperature is inversely proportional to its electrical resistivity. Since it is relatively intricate to measure the thermal conductivity, the measurement of electrical resistivity can be used as an indirect tool to deduce the thermal conductivity. The electrical resistivity at very low temperatures is mainly dictated by crystal defects and impurities and is termed as the residual resistivity. A very pure material would thus have a low residual resistivity (high RRR) and high thermal conductivity. The RRR is defined as the ratio between the resistances of the sample at room temperature (~300 K) and at temperature just above its superconducting transition temperature (~10 K).

In the RRR measurement set-up, the sample is introduced in the vapour column of liquid helium Dewar by a specially made test insert. The insertion depth is varied to change the temperature of the sample. The sample, along with temperature sensors, is sandwiched between two copper cover plates to minimize the temperature gradient along the sample. The sample is electrically insulated from its copper base by a 0.1mm thick *kapton* tape. The complete assembly is encapsulated by wrapping a 100 μm aluminium foil to ensure that upcoming helium vapour does not come in direct contact with the sample and temperature sensor, thus enabling stable measurements of temperature and resistance.

The resistance measurement is performed using the standard four-wire method. Current is fed by a constant current source (Lakeshore Cryo-tronics-120) and voltage is read through Keithley 182 voltmeter. Thermo-electric and galvanic voltages are eliminated by averaging voltage readings obtained by alternating the current polarities during each measurement. In order to keep the signal to noise ratio as high as possible, the external noise is reduced by using suitable filtering, using twisted pair of shielded conductors and unified grounding scheme. Current levels are kept as low as possible in pulsed mode to avoid intrinsic ohmic heating of sample and temperature drift. The current density through the sample is maintained between 0.001A/mm^2 to 0.1 A/mm^2 . Sufficient time is provided before each measurement to ensure stability and uniformity of temperature across the sample.

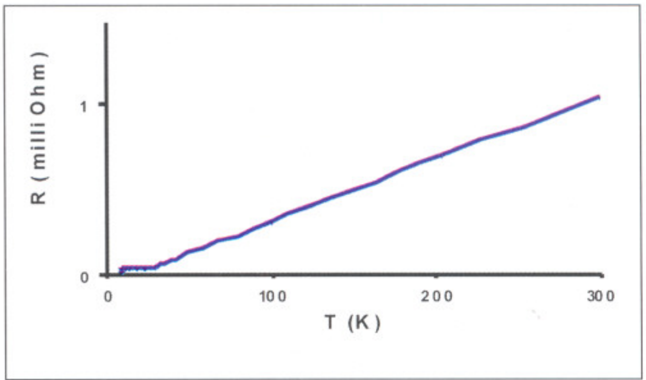

Fig. A.5.1: Temperature dependence of resistance of Nb Sample obtained from NFC, Hyderabad

The system calibration was done by comparing RRCAT measurements with those of samples from Fermi Lab and CERN (Table A.5.1). The repeatability of the apparatus was checked by re-measuring the samples after exposing to a large number of temperature cycles (Table A.5.2).

Table A.5.1: Calibration and measurement results

Source	Sample ID	Reported RRR	RRCAT Results	Deviation %
FNAL	Dec-07/04	364	367.7	1.09
FNAL	Dec-07/15	339	323.2	4.66
CERN	CERN/2	166	162.1	-2.34
CERN	CERN/4	189	187.9	- 0.58
BARC	K2/A/01	---	220	---
NFC	Nb/IC4/01	---	49	---
RRCAT	Nb/IMP	---	403	---

Table A.5.2:Repeatability test results

Source	Sample ID	Reported RRR	Test 1	Test 2	Rept. %
FNAL	Dec07/04	364	367.7	366.7	0.27
FNAL	Dec07/05	325	315.2	322.2	2.22
CERN	CERN/05	162	160	164.1	2.56
CERN	CERN/13	146	143	144.5	1.05
NFC	Nb/IC1/01	---	24.23	24.36	0.54
NFC	Nb/IC4/01	---	48.63	49.19	1.15
RRCAT	Nb/IMP	---	403	396.3	1.66

In conclusion, a facility for measuring the RRR of superconducting Nb has been set up. The results obtained from this apparatus are comparable within ± 5% of the reported values on known samples. The system repeatability is within 3% of the measured value of RRR. The measurement facility is operational and is being routinely used.

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A.6: Development of high reflectance Mo/Si soft x-ray multilayer mirror for normal incidence applications.

X-ray multilayer (ML) is a periodic arrangement of alternating layers of high-Z and low-Z elements. Mo/Si ML is one of the efficient mirror in the soft x-ray region at wavelengths above the Si L-edge (124 Å) due to high reflectivity. The surface and interfaces of Mo/Si have been studied extensively due to technological application such as lithography, astronomy, x-ray microscopy and spectroscopy. X-ray Optics Section of RRCAT has been fabricating high reflectance Mo/Si multilayers for developing Schwarzschild microscope for soft x-ray reflectivity / fluorescence beam-line on Indus-2 Synchrotron Radiation (SR) Source and polarimeter for soft x-ray polarization measurements on Indus-1 / Indus-2.

Mo/Si MLs were fabricated on Si wafer (roughness ~4 Å) using custom designed DC / RF magnetron sputtering system. The sputtering system has two rectangular cathodes of 500 mm length and 100 mm width. The sputtering was done using argon ion at a pressure of $3x10^{-3}$ mbar. The base vacuum in the chamber was ~2 10^{-8} mbar before starting the process. Substrates were cleaned using ion etching gun mounted in the load lock chamber. The deposition rate for Mo was ~4 Å/sec and for Si 0.3 Å/sec. Soft x-ray measurements were carried out on reflectometry beam-line at Indus-1 synchrotron radiation source. The beam-line provides monochromatic photons in the wavelength range of 10-300 eV, using a toroidal grating monochromator with a resolving power of 200-500.

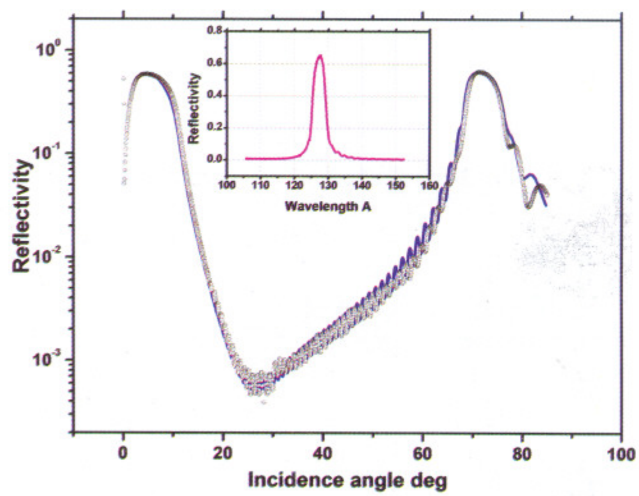

Fig. A.6.1: High reflactance multilayer mirror developed for normal incidence soft x-ray application with d=69Å, N=65 layer pairs, shows a 63% reflectivity. Continous line represents fitted curve and open circles are measured data points. In inset wavelength versus reflectivity curve measured at 72.5° deg incidence angle is shown.

Figure A.6.1 shows angle versus reflectivity scan of Mo/Si ML with d=69 Å and N=65 layer pairs at a wavelength of 127 Å. The multilayer gives 63% reflectivity at an incidence angle of 71°. The wavelength versus reflectivity scan, shown in the inset, was measured at a Bragg angle of 72.5°. Due to accurate control over individual layer thicknesses in the multilayer stacks, all the secondary oscillation fringes are measured. The multilayer with suitable varying periodicity will be used for potential application in the wavelength range of 126 -150 Å. The reflectivity performance achieved is in good agreement with the experimental values reported by different laboratories across the world.

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