Munich Cryogenic Detector Development 1995

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At the Technical University of Munich and the Max Planck Institute of Physics we are developing cryogenic detectors for the detection of small deposited energies, for example from the elastic scattering of WIMP dark matter particles, or the absorption of X-rays. Together with the University of Oxford and the Laboratori Nazionali del Gran Sasso we are preparing the CRESST experiment which uses our detectors to search for WIMP dark matter. This preprint contains reports of our work which we have presented at the Sixth International Workshop on Low Temperature Detectors (LTD-6) in Beatenburg/Interlaken, Switzerland, 28 Aug. – 1 Sept. 1995. This work has been supported in part by the “Sonderforschungsbereich 375 für Astroteilchenphysik” and the EU ERBCHRXCT930341 Network on Cryogenic Detectors.

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Progress on Fabrication of 
Iridium-Gold Proximity-Effect Thermometers

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Abstract

Iridium-gold proximity-effect bilayers with critical temperatures between 20 mK and 100 mK are made for use as superconducting phase transition thermometers for low temperature calorimeters. The reproducibility of the fabrication process of the iridium and gold films is discussed.

We have been developing massive cryogenic detectors [1, 2, 3, 4, 5] which use a superconducting phase transition thermometer evaporated directly onto a dielectric crystal. The energy deposited in the crystal by a single particle interaction is measured via the resulting temperature rise in the thermometer. The device is operated within the superconducting-to-normal transition of the thermometer, where a small temperature rise ΔT of the thermometer leads to a relatively large rise ΔR of its resistance. For a good temperature resolution a steep transition is required.

A low operating temperature increases the size of the signal but limits the choice of superconductor. We need critical temperatures still accessible with commercial ³He/⁴He dilution refrigerators. These requirements can be met by films of pure tungsten (Tc = 15 mK) [3, 5] and by films of iridium overlaid with gold, using the proximity effect to reduce the Tc of the combination below that of pure iridium (Tc = 112 mK). Detectors made with W and with Ir-Au thermometers on 32 g sapphire crystals have achieved comparable resolutions for 6 keV X-rays (160 eV [3] and 210 eV [2] FWHM, respectively). Ir-Au thermometers have the advantage of adjustable operating temperatures between 20 mK and 100 mK, allowing for more flexibility in the choice of the cryogenic equipment. Reproducible fabrication of these Ir-Au bilayers is the subject of this paper.

In our earlier work [4] iridium and gold films were produced by electron-beam evaporation in a high vacuum system. Tantalum shadow masks were used to produce films at

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desired places on the sapphire substrates. A water-cooled quartz crystal was used to monitor the deposited thickness during evaporation. Usually the iridium film was deposited first, with the substrate heated to between 500°C and 600°C. To monitor the film quality, we also evaporated diagnostic single layers of iridium and gold during the deposition of the bilayer.

The film thickness measured after evaporation by mechanical means deviated up to 10% from the desired thicknesses (700-1500 Å for iridium and 400-1000 Å for gold). A single film could vary in thickness by up to 50 Å along its length. The film quality in terms of crystal structure and impurities was described by the residual resistivity ratio RRR ≡ ρ₃₀₀K/ρ₄K. The value of the RRR of the iridium film varied between 1.3 and 5.4.

Critical temperatures of the iridium film and the Ir-Au bilayer were measured in a ³He/⁴He dilution refrigerator. We produced Ir-Au bilayers with Tc's as low as 33 mK. Data was compared to a model [4] derived from the de Gennes-Werthamer theory which describes the reduced Tc as a function of the thicknesses of the iridium and gold films, the RRR of the iridium film and the Tc of the single iridium film, which can be affected by impurities.

The model was used to predict the Tc dependence of the bilayer on the film parameters. A film thickness control of ±3% and a RRR of 8 ± 3 should yield an absolute error in Tc of below 3 mK for Tc's of ~34 mK and iridium film thicknesses of ~1000 Å (Fig. 1).

To achieve this desired reproducibility in the film parameters, the evaporation system has been extensively modified. The films are now produced by a three crucible electron-beam evaporator at a pressure of 1 × 10⁻⁹ mbar. Films are evaporated onto the epi-polished surface of the 10 x 20 x 0.5 mm³ sapphire crystals. We use 99.999% purity iridium single crystals (RRR ~ 400) from BEC Breznar and 99.999% purity gold wire from Degussa as evaporation materials. The substrate heater and holder were made out of tantalum and sapphire to avoid magnetic contamination of the films. The maximum substrate temperature is 730°C. Special attention was paid to thickness control during evaporation. The quartz crystal was placed closer to the sub-
strates (4 cm now compared to 7 cm in the old system). The distance between substrates and evaporator was increased from 15 cm to 32 cm to improve film thickness homogeneity while still depositing at a reasonable rate. The whole inner setup is surrounded by a radiation shield cooled with liquid nitrogen. A load lock allows us to maintain the vacuum in the evaporation chamber for more than one evaporation run to assure equal conditions. An automatic system controls the degasing of the evaporation material, moving the shutter, and adjusting the evaporation rate.

TRFA (Total Reflection X-ray Fluorescence Analysis) of the BEC crystal surface before deposition showed contamination of Fe and Ni at the order of $10^{14}$ atoms/cm$^2$, which corresponds to almost one monolayer. To reduce this surface contamination, several cleaning procedures have been investigated. A combination of chemicals was found that reduced the contamination of Fe and Ni to the order of $3 \times 10^{12}$ atoms/cm$^2$.

Substrates were degased before deposition at a temperature 50$^\circ$C higher than the evaporation temperature for 30 minutes. Films were made at varying substrate temperatures from 350$^\circ$C to 630$^\circ$C with evaporation rates of 0.1 Å/s for iridium and 1 Å/s for gold. After deposition films were structured via photolithography and sputter etching for film thickness analysis and RRR measurement.

With this new setup and fabrication procedure we report film thickness control during evaporation of ±3% for the iridium and gold films. The film thickness varies less than 15 Å at different positions on the film. The measured RRR of the iridium films is about 2 for substrate temperatures of 300$^\circ$C and about 8 for 630$^\circ$C. A variation of $\Delta$RRR<3 for fixed substrate temperature is estimated (with small statistics). We expect that the reproducible production of Ir-Au bilayers with $T_c$'s as low as 20 mK will be possible soon.

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[5] P. Ferger, et al., these proceedings