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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.

Contents:

Measurement of the tunnel rate in S-I-S' tunnel junctions as function of bias voltage H. Kraus, et al.

Cryogenic Particle Detectors with Supeconducting Phase Transition Thermometers P. Ferger, et al.

Progress on Fabrication of Iridium-Gold Proximity-Effect Thermometers J. Höhne, et al.

Status and Low Background Considerations for the CRESST Dark Matter Search M. Bühler, et al.

Cross section of low mass neutralino dark matter in the Unconstrained Minimal Supersymmetric Standard Model, A. Gabutti, et al.

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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 $(T_c=15 \,\mathrm{mK})$ [3, 5] and by films of iridium overlaid with gold, using the proximity effect to reduce the T_c of the combination below that of pure iridium $(T_c=112\,\mathrm{mK})$. Detectors made with W and with Ir-Au thermometers on 32g 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

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 resitivity ratio RRR $\equiv \rho_{300K}/\rho_{4K}$. 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 ${}^{3}\text{He}/{}^{4}\text{He}$ dilution refrigerator. We produced Ir-Au bilayers with T_c 's as low as $33\,\text{mK}$. Data was compared to a model [4] derived from the de Gennes-Werthamer theory which descibes the reduced T_c as a function of the thicknesses of the iridium and gold films, the RRR of the iridium film and the T_c of the single iridium film, which can be affected by impurities.

The model was used to predict the T_c dependence of the bilayer on the film parameters. A film thickness control of $\pm 3\%$ and a RRR of 8 ± 3 should yield an absolute error in T_c of below $3\,\mathrm{mK}$ for T_c 's of $\sim 34\,\mathrm{mK}$ and iridium film thicknesses of $\sim 1000\,\mathrm{\AA}$ (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 $1\times 10^{-9}\,\mathrm{mbar}$. Films are evaporated onto the epi-polished surface of the $10\times 20\times 0.5\,\mathrm{mm^3}$ sapphire crystals. We use 99.999% purity iridium single crystals (RRR ~ 400) from BEC Breznikar 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 $730^{\circ}\mathrm{C}$. Special attention was paid to thickness control during evaporation. The quartz crystal was placed closer to the sub-

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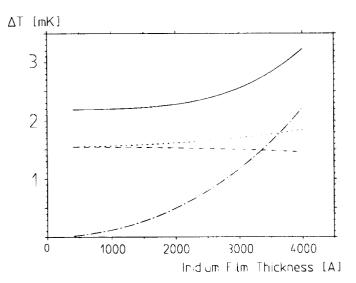


Figure 1: The expected absolute reproducibility ΔT_c of an Ir-Au bilayer designed to have $T_c{=}34\,\mathrm{mK}$ calculated from the model using an average RRR of 8 for the iridium film. The dotted and dashed lines are the contributions of the film thickness variation of $\pm 3\%$ for the iridium and gold films. The dashed-dotted line shows the contribution of $\Delta RRR{=}3$. The solid line is is the total T_c variation, calculated by adding these three contributions quadradically.

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 evaporation showed contamination of Fe and Ni at the order of 10^{14} atoms/cm², 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 3×10^{12} atoms/cm².

Substrates were degased before deposition at a temperature 50°C higher than the evaporation temperature for 30 minutes. Films were made at varying substrate temperatures from 350°C to 630°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 $\pm 3\%$ 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° C and about 8 for 630° C. A variation of Δ RRR<3 for fixed substrate temperature is estimated (with small statis-

tics). 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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