Treatments To Produce Stabilized Aluminum Mirrors for Cryogenic Uses

Selected heat treatments are performed between and after fabrication steps.

Goddard Space Flight Center, Greenbelt, Maryland

Five metallurgical treatments have been tested as means of stabilizing mirrors that are made of aluminum alloy 6061 and are intended for use in cryogenic applications. Aluminum alloy 6061 is favored as a mirror material by many scientists and engineers. Like other alloys, it shrinks upon cool-down from room temperature to cryogenic temperature. This shrinkage degrades the optical quality of the mirror surfaces. Hence, the metallurgical treatments were tested to determine which one could be most effective in minimizing the adverse optical effects of cool-down to cryogenic temperatures. Each of the five metallurgical treatments comprises a multistep process, the steps of which are interspersed with the steps of the mirror-fabrication process. The five metallurgical-treatment/fabrication— process combinations were compared with each other and with a benchmark fabrication process, in which a mirror is made from an alloy blank by (1) symmetrical rough machining, (2) finish machining to within 0.006 in. (≈ 0.15 mm) of final dimensions, and finally (3) diamond turning to a mirror finish. Two specimens — a flat mirror and a spherical mirror — were fabricated in each case. The blanks for all the specimens were cut from the same plate of aluminum alloy 6061-T651. (The suffix "T651" denotes a stress-relieving treatment that involves reducing residual stresses by mechanical stretching of the previously untreated alloy.) Of the five metallurgical-treatment/fabrication-process combinations tested, the one found to exert the greatest stabilizing effect comprises the following ten steps:

1. Rough machining.
2. Solution heat treatment at a temperature of 985 °F (≈ 529 °C).
3. Quench within 15 s in a solution of 28 percent UCON Quenchant A (or an equivalent aqueous quenching liquid) at a temperature of 90 °F (≈ 32 °C).
4. Uphill quench: Allow to reach room temperature, slowly dip into a tank of liquid nitrogen followed by rapid immersion in a tank of boiling water.
5. Age at 350 °F (≈ 177 °C).
6. Finish machine leaving about 0.006 in. (0.15 mm) for polishing.
7. Age again as in step 5.
8. Three thermal cycles with heating and cooling rates not to exceed 3 °F/min (1.7 °C/min) as follows: Cool to -310 °F (-190 °C), hold for 30 min, heat to room temperature, hold for 15 min, heat to 300 °F (150 °C), hold for 15 minutes, and cool to room temperature.
9. Diamond turning/polishing.
10. Three thermal cycles as in step 7.

Separate tests showed that forged AL6061 with the same processing method would yield slightly better mirrors than those made from AL6061-T651 plates.

This work was done by Wahid Zewari, Michael Barthelmy, and Raymond Ohl of **Goddard Space Flight Center**. *Further information is contained in a TSP (see page 1).*

Inquiries concerning rights for the commercial use of this invention should be addressed to the Patent Counsel, Goddard Space Flight Center, (301) 286-7351. Refer to GSC-14736-1.

Making AlN_x Tunnel Barriers Using a Low-Energy Nitrogen-Ion Beam

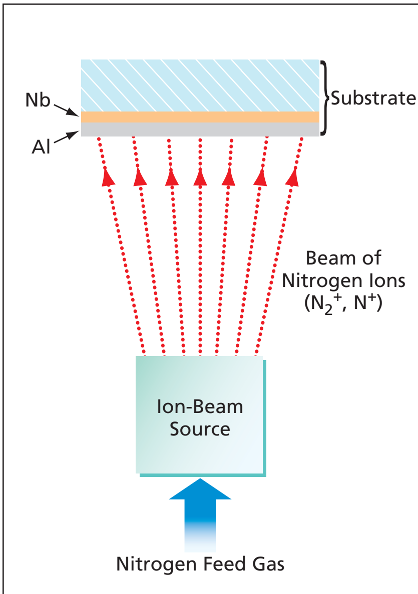
Ion-beam parameters can be controlled to optimize properties of AlN_x layers.

NASA's Jet Propulsion Laboratory, Pasadena, California

A technique based on accelerating positive nitrogen ions onto an aluminum layer has been demonstrated to be effective in forming thin (<2 nm thick) layers of aluminum nitride (AlN_x) for use as tunnel barriers in Nb/Al-AlN_x/Nb superconductor/insulator/superconductor (SIS) Josephson junctions. AlN_x is the present material of choice for tunnel barriers because, to a degree greater than that of any other suitable material, it offers the required combination of low leakage current at high current density and greater thermal stability.

While ultra-thin AlN films with good thickness and stoichiometry control are easily formed using techniques such as reactive molecular beam epitaxy and chemical vapor deposition, growth temperatures of 900 ºC are necessary for the dissociative adsorption of nitrogen from either nitrogen (N_2) or ammonia (NH_3). These growth temperatures are prohibitively high for the formation of tunnel barriers on Nb films because interfacial reactions at temperatures as low as 200 to 300 ºC degrade device properties. Heretofore, deposition by reactive sputtering and nitridation of thin Al layers with DC and RF nitrogen plasmas have been successfully used to form AlN barriers in SIS junctions. However, precise control over critical current density J_c has proven to be a challenge, as is attaining adequate process reproducibility from system to system.

The present ion-beam technique is an alternative to the plasma or reactive sputtering techniques as it provides a highly controlled arrival of reactive species, independent of the electrical conditions of the substrate or vacuum chamber. Independent and accurate control of parameters such as ion en-



The **Beam of Nitrogen Ions** impinges on the aluminum substrate surface layer, forming an ultra-thin layer of AlN_x.

ergy, flux, species, and direction promises more precise control of film characteristics such as stoichiometry and thickness than is the case with typical plasma processes. In particular, the background pressure during ion-beam nitride growth is 2 or 3 orders of magnitude lower, minimizing the formation of compounds with contaminants, which is critical in devices the performance of which is dictated by interfacial characteristics. In addition, the flux of incoming species can be measured *in situ* using ion probes so that the dose can be controlled accurately.

The apparatus used in the present ion-beam technique includes a vacuum chamber containing a commercial collimated-ion-beam source, a supply of nitrogen and argon, and an ion probe for measuring the ion dose. Either argon or nitrogen can be used as the feed gases for the ion source, depending on whether cleaning of the substrate or growth of the nitride, respectively, is desired. Once the Nb base electrode and Al proximity layer have been deposited, the N_2 gas line to the ion beam is vented and purged, and the ion-source is turned on until a stable discharge is obtained. The substrate is moved over the ion-beam source to expose the Al surface layer to the ion beam (see figure) for a specified duration for the formation of the nitride tunnel barrier. Next, the Nb counter-electrode layer is deposited on the nitride surface layer. The Nb/Al-AlN_x/Nb-trilayer-covered substrate is then patterned into individual devices by use of conventional integrated-circuit processing techniques.

A wide parameter space was investigated over which devices were fabricated reproducibly and with high quality. The hysteretic nature of the current-voltage characteristic along with the high subgap ratio indicate the incident nitrogen ions chemically reacted with the Al layer as expected, to form a continuous AlN_x barrier. Chemical analysis of the barrier performed using x-ray photoelectron-spectroscopy confirmed the presence of AlN_x. Critical current density J_c ranged from 550 to 9,400 A/cm^2 with subgap-to-normal resistance ratios ranging from 50 to 12.6. The J_c was found to decrease with increasing dose and increasing beam energy. The run-to-run reproducibility was determined to be very good. The spatial variation of the ion current density was also measured and correlated with J_c over a 76-mm Si wafer. The junctions were also found to be stable on annealing up to temperatures of 250 °C. This technique could be applied to form other metal nitrides at room temperatures for device applications where a high degree of control is desired.

This work was done by Anupama Kaul, Alan Kleinsasser, Bruce Bumble, Henry LeDuc, and Karen Lee of Caltech for **NASA's Jet Propulsion Laboratory**. *Further information is contained in a TSP (see page 1). NPO-41297*

Making Wide-IF SIS Mixers With Suspended Metal-Beam Leads

Devices are fabricated on SOI substrates by use of silicon-micromachining techniques.

NASA's Jet Propulsion Laboratory, Pasadena, California

A process that employs silicon-on-insulator (SOI) substrates and silicon (Si) micromachining has been devised for fabricating wide-intermediate-frequency-band (wide-IF) superconductor/insulator/superconductor (SIS) mixer devices that result in suspended gold beam leads used for radio-frequency grounding. The mixers are formed on 25-μm-thick silicon membranes. They are designed to operate in the 200 to 300 GHz frequency band, wherein wide-IF receivers for tropospheric-chemistry and astrophysical investigations are necessary.

The fabrication process can be divided into three sections:

1. The front-side process, in which SIS devices with beam leads are formed on a SOI wafer;
2. The backside process, in which the SOI wafer is wax-mounted onto a carrier wafer, then thinned, then partitioned into individual devices; and
3. The release process, in which the individual devices are separated using a lithographic dicing technique.

The total thickness of the starting 4-in. (10.16-cm)-diameter SOI wafer includes 25 μm for the Si device layer, 0.5 μm for the buried oxide (BOX) layer, and 350 μm the for Si-handle layer. The front-side process begins with deposition of an etch-stop layer of SiO_2 or AlN_x, followed by deposition of a Nb/Al-AlN_x/Nb trilayer in a load-locked DC magnetron sputtering system. The lithography for four of a total of five layers is performed in a commercial wafer-stepping apparatus. Diagnostic test dies are patterned concurrently at certain locations on the wafer, alongside the mixer devices, using a different mask set. The conventional, self-aligned lift-off process is used to pattern the SIS devices up to the wire level.

The beam-leads are formed as extensions from the SIS devices by using a bilayer lift-off process with poly(methyl methacrylate) [PMMA] and photoresist. After defining the beam-leads, the interfacial layer between the PMMA and photoresist is etched in an oxygen plasma. Ultraviolet irradiation is used to expose the PMMA, which is then developed in chlorobenzene. The wafer is then placed in the sputtering system, where a seed layer of Nb/Au is deposited to enhance adhesion. The Au beam leads are grown to the desired thickness in an electron-beam evaporation system. After deposition, the unwanted gold is easily removed by lift-off in acetone.