LBL-34447
UC-404

Lawrence Berkeley Laboratory
UNIVERSITY OF CALIFORNIA

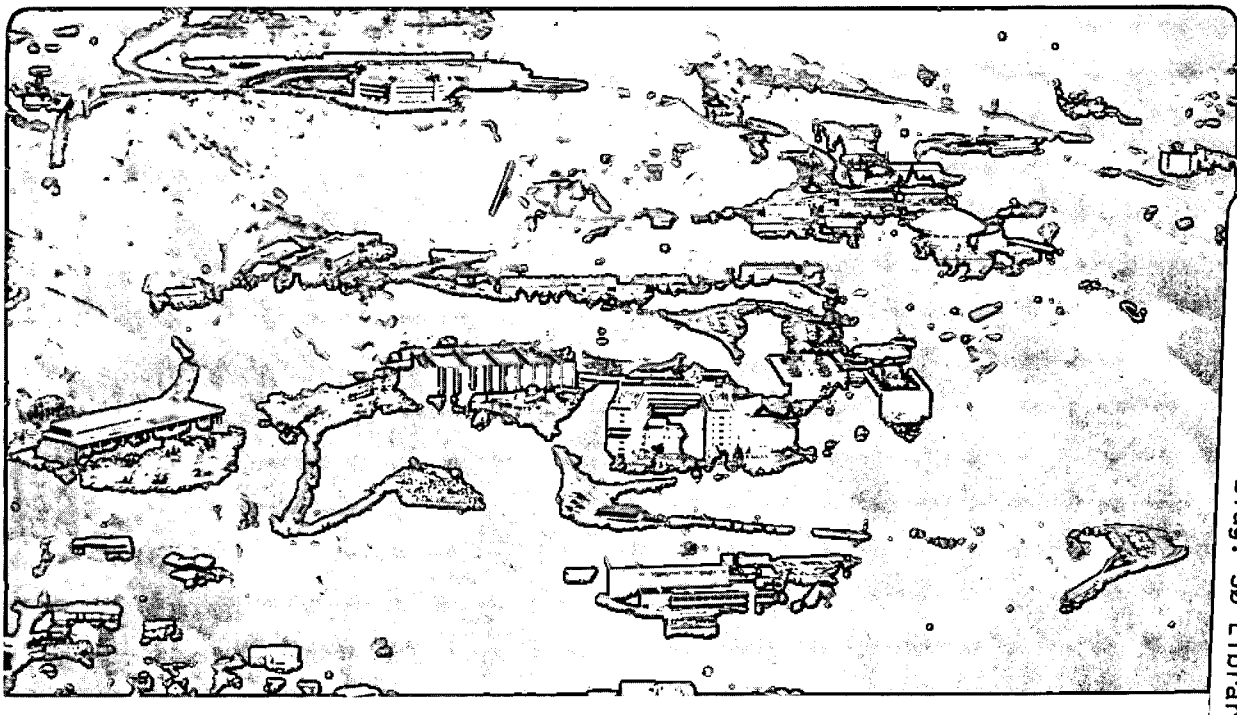
Engineering Division

Presented at the Fifth International Workshop on Low Temperature Detectors, Berkeley, CA, July 29–August 3, 1993, and to be published in the Proceedings

Ge-Au Eutectic Bonding of Ge {100} Single Crystals

W. Knowlton, K.M. Itoh, J. Beeman,
J. Emes, D. Loretto, and E.E. Haller

August 1993



REFERENCE COPY
Does Not
Circulate
Bldg. 50 Library.
LBL-34447
Copy 1

Prepared for the U.S. Department of Energy under Contract Number DE-AC03-76SF00098

DISCLAIMER

This document was prepared as an account of work sponsored by the United States Government. While this document is believed to contain correct information, neither the United States Government nor any agency thereof, nor the Regents of the University of California, nor any of their employees, makes any warranty, express or implied, or assumes any legal responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by its trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or the Regents of the University of California. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof or the Regents of the University of California.

LBL-34447

Ge-Au Eutectic Bonding of Ge {100} Single Crystals

W. KNOWLTON, K.M. ITOH, J. BEEMAN, J. EMES, D. LORETTO AND E.E. HALLER

ENGINEERING DIVISION
Lawrence Berkeley Laboratory
University of California
Berkeley, California 94720

AUGUST 1993

This work was supported in part by the U.S. National Science Foundation through the Center for Particle Astrophysics at UC Berkeley under contract ADT-8809616, and by NASA contract W17605 through interagency agreement with the U.S. Department of Energy under Contract No. DE-AC03-76SF00098.

Ge-Au Eutectic Bonding of Ge {100} Single Crystals

W.B. Knowlton*, K.M. Itoh*,†, J.W. Beeman*, J.H. Emes*, D. Loretto*, and E.E. Haller*,†

**Lawrence Berkeley Laboratory; Berkeley, CA 94720 USA and*
†University of California at Berkeley, Berkeley, CA 94720 USA

We present preliminary results on the eutectic bonding between two {100} Ge single crystal surfaces using thin films of Au ranging from 900Å/surface to 300Å/surface and Pd (10% the thickness of Au). Following bonding, plan view optical microscopy (OM) of the cleaved interface of samples with Au thicknesses ≤ 500Å/surface show a eutectic morphology more conducive to phonon transmission through the bond interface. High resolution transmission electron microscopy (HRTEM) cross sectional interface studies of a 300Å/surface Au sample show <100> epitaxial growth of Ge. In sections of the bond, lattice continuity of the Ge is apparent through the interface. TEM studies also reveal <110> heteroepitaxial growth of Au with a Au-Ge lattice mismatch of less than 2%. Eutectic bonds with 200Å/surface Au have been attained with characterization pending. An optical polishing technique for Ge has been optimized to insure intimate contact between the Ge surfaces prior to bonding. Interferometry analysis of the optically polished Ge surface shows that surface height fluctuations lie within ±150Å across an interval of 1mm. Characterization of phonon transmission through the interface is discussed with respect to low temperature detection of ballistic phonons.

1. INTRODUCTION

Currently, the search for dark matter to substantiate the theory that, in spacetime geometry, our universe is "flat" is of high priority. Researchers at the University of California at Berkeley have demonstrated that using composite phonon-mediated detectors, basically low temperature semiconductor thermometers, may be a viable means of detecting a particular type of dark matter at milliKelvin temperatures.[1-3] The composite phonon-mediated detector is comprised of two components: the "antenna" and the thermistor (see Fig. 1). The antenna is an ultra pure single crystal semiconductor (in this case, Ge with $\sim 10^{10} cm^{-3}$ electrically active impurities) which is the incident particle absorbing and energy transporting medium. The antenna is bonded to the thermistor: a smaller (here, about 1/2000th the volume of the antenna) doped semiconductor (again Ge) which acts as the detecting medium.

At low temperatures, energy deposited by incident dark matter in the antenna will result in the creation of acoustic phonons via a down conversion of optical phonons.[4,5] Due to the purity of the Ge antenna, the phonons will propagate ballistically (i.e., without scattering) through the pure single crystal Ge to the thermistor. The thermistor "traps" the phonons by scattering processes which thermalize the phonons. The thermalized phonons increase the temperature of the material which

in turn leads to a decrease in resistivity. From the decrease in resistivity, the energy deposited by the incident particle can be determined.[6]

The use of composite bolometers in low temperature applications has created the need for a means of adhering thermistors to the antenna with minimal phonon scattering at the bonded interface. The boundary frequency of acoustic phonons in Ge is on the order of 10^{10} Hz.[7,8] Because the acoustic phonon, which propagates at the velocity of sound, has a wavelength on the order of 500Å, interfaces > 500Å will scatter incident phonons. Therefore, in this study, we focus on the experiments involved in developing Au-Ge eutectic bonds transparent to ballistic phonons and the characterization of the eutectic interfaces.

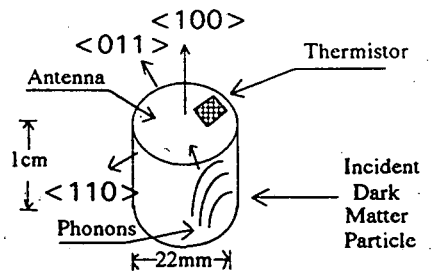


Fig. 1: Composite Phonon-Mediated Detector

The Au-Ge eutectic bonding method is attractive because it is a low temperature process. The eutectic temperature, T_e, of the Au-Ge system is 361°C while the melting points, T_m, of Au and Ge are respectively 1064°C and 938°C.[9] Thus, annealing temperatures, which are about 50°C above T_e, are far below the T_m's of the materials. Since the diffusivity of Au is very small in Ge ($<10^{-15}m^2s^{-1}$ at 600°C), and the solubility of Au in Ge is very low ($10^{15}cm^{-3}$ at 550°C), the phase separated Ge will be practically free of Au.[10,11] This significantly decreases the addition of impurities into the bulk material.

In this paper, we report on structural studies of Au-Ge eutectic bonding of two single crystal (100) Ge wafers with respect to minimizing phonon scattering at the eutectic interface.

2. EXPERIMENTAL PROCEDURE

A Czochralski grown, low dislocation density Ge ingot was mounted on a graphite block. Two 1mm thick, 3.5cm diameter (100) Ge wafers were cut with an inside diameter (ID) diamond saw. With the Ge wafers still attached to the graphite block, notches were scribed into the wafers to retain the wafers orientation. To obtain an atomically flat and smooth surface morphology, the Ge wafers were individually lapped and polished. Both sides of each wafer were lapped using a 9μm Al_2O_3 grit solution. The bonding surface of each wafer was polished in a 7:3:1 solution of de-ionized water, alkaline SiO_2 colloidal suspension, and 30% H_2O_2. The polished surfaces of the wafers were placed together and the notches were aligned. In this configuration, the wafers were mounted on a graphite block and cut using an ID diamond saw producing $3x4mm^2$ rectangular bonding pairs. Thin films of Pd and Au were electron beam deposited on the polished surface of the two rectangular Ge samples. The Pd film was 10% the thickness of the Au film and was used to eliminate residual oxides and to decrease the surface tension of the Au on Ge during Au deposition. The Ge samples were placed in a clamping apparatus such that the thin films were sandwiched between the two samples

and were placed in a tube furnace. They were annealed at 415°C for two hours then cooled at a rate of ~22°C/hr to 200°C. Several of the bonded pairs were cleaved at the interface or through the interface for plan view and cross sectional studies, respectively.

3. CHARACTERIZATION

Several characterization techniques including He/Ne laser interferometry, optical microscopy, and transmission electron microscopy (TEM) were utilized in this study. Optical microscopy was employed to examine the surface morphology of the cleaved eutectic bonds. TEM was used to examine the surface morphology, crystallinity, structural continuity, and lattice mismatch of the eutectic interface.

In order to achieve a structurally intact Au-Ge eutectic bond such that intimate contact is made throughout the interface, the surface morphology of the Ge faces on which Pd and Au is to be deposited need to be extremely smooth and as flat as possible. Methods for attaining atomically flat Ge wafers were optimized using He/Ne laser interferometry. Problems in the mounting procedure were determined and circumvented through analysis of interferograms. The result of such an analysis is depicted in the interferogram in Fig. 2. The Ge wafer is located in the center of the surrounding polycrystalline Ge "spacers" used to enhance the lapping and polishing process. As shown, the 3.5cm diameter Ge wafer is within one Newtonian ring. This reveals that over the radius of the wafer, the surface height change is < 3164Å ($\lambda_{He/Ne}$=6328Å). That is, over an interval of 1mm, the change in surface height is < 200Å indicating an atomically flat surface. Secondly, the symmetric appearance of the fringe denotes a smooth surface morphology.

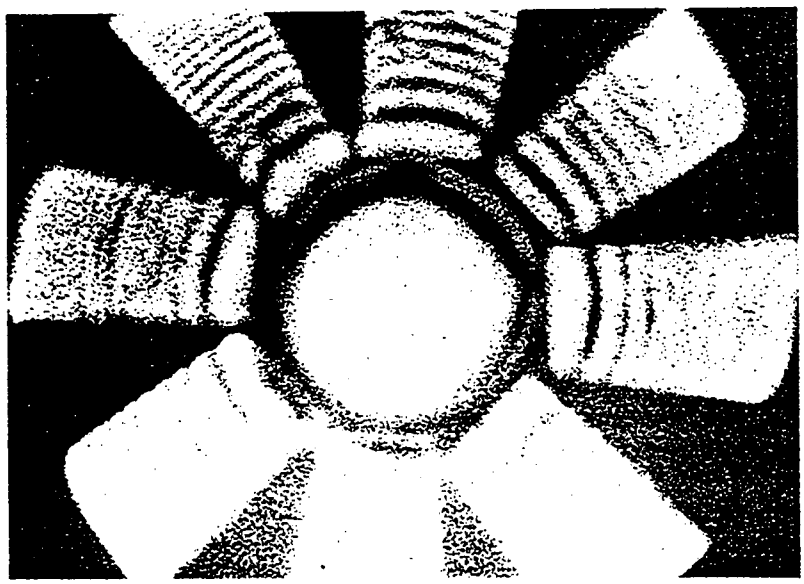


Fig. 2. He/Ne laser interferogram of an optically polished 3.5cm diameter Ge wafer.

After annealing, the cleaved surface morphology of eutectic bonds with differing Pd and Au thicknesses was examined using optical microscopy. Films of 900Å/surface (sample surface) and 700Å/surface gave rise to Ge area coverages of 20% and 30% respectively. The dendritic eutectic structure was retained with films thicker than 500Å/surface as seen in Fig. 3 (a). However, for films of 500Å/surface or less, the dendritic eutectic morphology was replaced by the formation of single crystal <110> ellipsoidal Au precipitates covering only 20% to 30% of the interface. The remaining area was single crystal <100> Ge (see Fig.3 (b)). The structure of the Au islands and single crystal Ge between was independently determined by TEM selected area diffraction patterns. In this case, phonon attenuation is drastically reduced due to the increase of Ge surface area and decrease of Au precipitates which scatter phonons.

Finally, phase contrast high resolution transmission electron micrographs (HRTEM) were taken of the interfacial cross section of a 300Å/surface sample. The HRTEM shown in Fig. 4 depicts Ge homoepitaxial growth across the interface. In other words, Ge lattice continuity through the interface is apparent. The pixels resembling atoms in Fig. 4 are Ge "dumb bells" and correspond to two Ge atoms. The top half of the micrograph is focused on the [110] zone axes. The bottom half of the micrograph is just slightly out of focus illustrating that less than a 1° misorientation occurs between the two Ge interfaces.

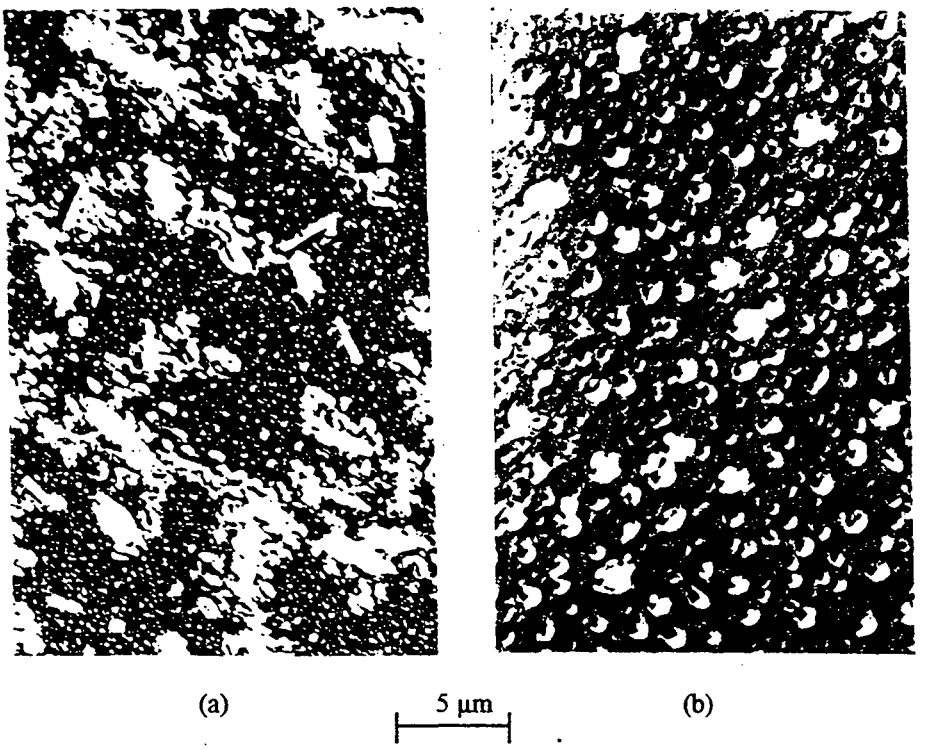
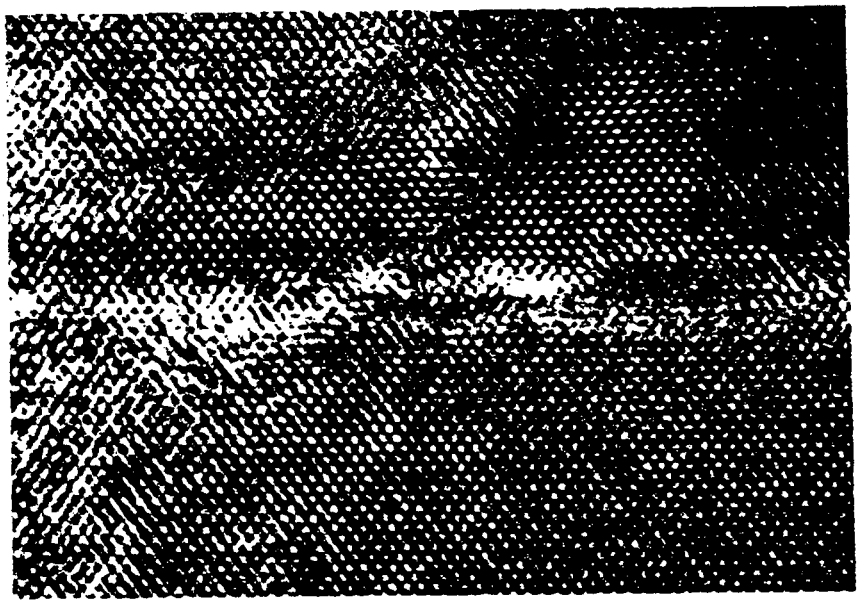


Fig. 3 Cleaved surface morphology of eutectic bonds with Pd and Au thickness: (a) 700Å/ surface and (b) 300Å/surface.



50Å

Fig. 4 High resolution transmission electron micrograph of the Ge-Au eutectic interfacial cross section with Pd and Au thickness 300Å/surface.

4. CONCLUSION

In this paper we present a structural analysis of the Au-Ge eutectic bond interface. The aim is to maximize the interfacial ballistic phonon transparency. The process which we developed should provide an interface which produces minimal stress, low amounts of impurities, and insure continuity. Atomically flat and smooth bonding surfaces were attained. A surface height change of < 200Å across a 1mm wafer interval after polishing was achieved. Scattering centers were minimized by the fact that the Ge interface coverage was increased and Au coverage was decreased when using thin films less than 500Å/surface. In sections of the interface, lattice continuity was attained due to the [001] homoepitaxial growth of Ge through the interface.

ACKNOWLEDGMENTS

We would like to thank Wendy Swider, Rodney Post, and Steve Rothway for their technical assistance. This work was supported in part by the US National Science Foundation through the Center for Particle Astrophysics at UC Berkeley under contract ADT-8809616, and by NASA contract W17605 through interagency agreement with the US Department of Energy Contract DE-AC03-76SF00098.

REFERENCES

[1] N. Wang, B. Sadoulet, T. Shutt, J. Beeman, E.E. Haller, A. Lange, I. Park, R. Ross, C. Stanton and H.Steiner, *IEEE Trans. in Nuclear Science* **NS 35**, 852 (1988).

[2] A. Cummings, N. Wang, T. Shutt, P. Barnes, J. Emes, Y. Giraud-Heraud, E.E. Haller, A. Lange, J. Rich, R. Ross, B. Sadoulet, G. Smith and C. Stubbs, *IEEE Trans. Nucl, Sci. Symp. Rec.* **1**, 209 (1990).

[3] T.A. Shutt, in *A Dark Matter Detector Based on the Simultaneous Measurement of Phonons and Ionization at 20 mK* (Ph.D. Thesis, Physics Dept., University of California at Berkeley, 1993).

[4] H.J. Maris, in *Phonon Scattering in Condensed Matter*, edited by A.C. Anderson and J.P. Wolfe (Springer-Verlag, New York, 1986), p. 404.

[5] N. Wang, J. Beeman, A.N. Cleland, A. Cummings, E.E. Haller, A. Lange, R. Ross, B. Sadoulet, H. Steiner, T. Shutt and F.C. Wellstood, *IEEE Transactions on Nuclear Science* **36**, 852 (1989).

[6] E.E. Haller, N.P. Palaio, M. Rodder, W.L. Hansen and E. Kreysa, in *Neutron Tranmutation Doping of Semiconductor Materials*, edited by R.D. Larrabee (Plenum, New York, 1984), p. 21.

[7] G. Nilsson and G. Nelin, *Phys. Rev. B* **3**, 364 (1971)

[8] S. Tamura, J.A. Shields and J.P. Wolfe, *Phys. Rev. B* **44**, 3001 (1991).

[9] H. Okamoto and T.B. Massalski, in *Binary Alloy Phase Diagrams*, edited by T.B. Massalski, J.L. Murray, L.H. Bennett and H. Baker (William W. Scott, Jr., Metals Park, 1984), p. 263.

[10] H. Bracht, N.A. Stolwijk and H. Mehrer, *Physics Review B* **43**, 14 (1991).

[11] A. Almazouzi, J. Bernardini, E.G. Moya, H. Bracht, N.A. Stolwijk and H. Mehrer, *J. Appl. Phys.* **70**, 1345 (1991).

LAWRENCE BERKELEY LABORATORY
UNIVERSITY OF CALIFORNIA
TECHNICAL INFORMATION DEPARTMENT
BERKELEY, CALIFORNIA 94720