Title

INTRUSION BONDING OF NICKEL AND ZIRCONIA

Permalink

https://escholarship.org/uc/item/8vx6k759

Authors

Reimanis, I.E.
Shinde, S.L.
Jonghe, L.C. De

Publication Date

1987-06-01

LBL-23651
Preprint

Lawrence Berkeley Laboratory
UNIVERSITY OF CALIFORNIA

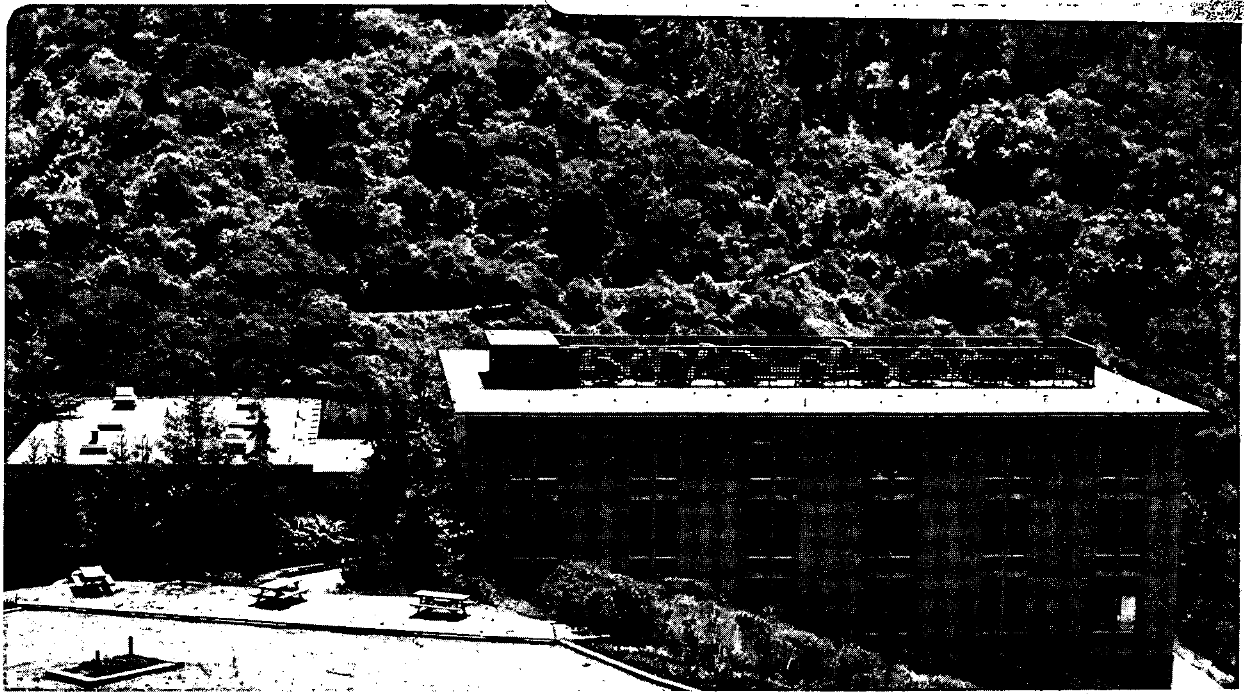
Materials & Chemical Sciences Division

Submitted to Journal of the
American Ceramic Society

INTRUSION BONDING OF NICKEL AND ZIRCONIA

I.E. Reimanis, S.L. Shinde,
and L.C. De Jonghe

June 1987



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

INTRUSION BONDING OF NICKEL AND ZIRCONIA

I.E.Reimanis*, S.L.Shinde**, and L.C.De Jonghe
Lawrence Berkeley Laboratory Materials and Chemical Sciences Division and
Department of Materials science and Mineral Engineering University of
California Berkeley, CA 94720

Abstract

Intrusion bonding of nickel to zirconia was achieved by hot pressing the zirconia powder onto Ni sheet, at pressures of about 55MN/m^2, and temperatures of about 1573K. The intrusion zone can be several hundred micrometers thick, providing a gradual transition from ceramic to metal. The most extensive intrusion occurred when pressure was applied late in the heating cycle, after the zirconia powder had coarsened. The parameters determining the extent of the intrusion bonding are discussed. The intrusion bonds are compared to diffusion bonds for which a discrete metal/ cerami interface was maintained. Vickers microindentation tests in the intrusion bond zone indicated that metal intrusion provided improved bonding.

1 Introduction

In many applications it is necessary to control the intermixing of metal with the material to which it is joined. Important examples are metal leads co-sintered in multilayer ceramic substrates, and thermal barrier coatings for turbine blades. In these applications, intrusion of the metal into the ceramic phase is deleterious to the proper functioning of the components. In other cases, intermixing is useful in improving the performance of metal/ ceramic junctions.[1]

The phenomenon of metal intrusion during the hot pressing of zirconia powder onto nickel sheet metal is reported here. The processing parameters are discussed that determine the degree of metal penetration during bonding.

---

* Present address: Materials Department, UC Santa Barbara, Santa Barbara, CA 93106.
**Present Address: IBM Yorktown, Thomas B.Watson Research Center,Yorktown Heights,NY10598

Two types of interfaces were produced: 1) powder hot pressed onto nickel sheet, giving an intrusion bond, and 2) presintered zirconia pressed onto nickel sheet at low stress, giving a diffusion bond.

2. Experimental

Zirconia, stabilized by 5.5 wt% or by 10.5 wt% calcia was obtained from the Magnesium Elektron Company for bonding to small, polished nickel disks 0.1 to 0.3 mm thick.

A first type of junction was fabricated by hot pressing zirconia powder directly onto the polished nickel disks, at pressures of 55 MN/m^2, at 1573K, in a cylindrical graphite die. The microstructure and the density of the zirconia could be varied by changing the time at temperature and the loading cycle of the hot pressing. The nickel intrusion zones were generally clearly distinguishable, with a fairly abrupt boundary between the metal-free zirconia and the intrusion zone. The depth of intrusion was determined from the position of this boundary.

A second type of junction was produced by pressing pre-sintered zirconia onto the nickel disks at temperature between 1273 an 1573K, with loads up to 7 MN/m^2.

Some junctions received additional anneals in air, at 1273K, for 1, 10, or 65 hours. The densities of the ceramics were determined by Archimedes' method and theoretical densities were determined assuming 100 % cubic zirconia with 5.5. or 10.5 wt% calcia.

The microstructures of the junctions were characterized by optical and scanning electron microscopy , and the mechanical integrity of the junctions was qualitatively evaluated by Vickers micro-indentation.

3. Results and Discussion

A. Intrusion Bonds

The extent of the nickel intrusion during the joining-hot pressing operation was determined by the temperature and by the time at which the full pressure of 55 MN/m^2 was applied. The average of the measured intrusion distances have been listed in Table 1. Perhaps contrary to expectations, the most extensive intrusion occured when the full pressure was applied late in the hot pressing cycle, as is evident from Table 1. When the pressure was applied early, immediately after the hot pressing temperature had been reached, as shown in Fig 1B, less extensive intrusion occured. Still less intrusion was found for joints where the full pressure was applied at low temperatures and held constant throughout, Fig 1C. Finally, almost no intrusion occured if the applied pressure was held at about 2 MN/m^2. The hot pressed densities of the metal- free zirconia ceramic of the joints increase with increasing length of time of the application of the full pressing load, see Table 1.

The dependence of the intrusion structure on the processing parameters may be understood by considering the plastic flow behavior of nickel metal, microstructural development of the sintering zirconia, and the densifciation rates. The rate at which nickel will intrude during hot pressing will be determined by flow stress of the nickel, the diameter of the intrusion channels, and wether or not the pores form an open network. While not accurately known here, the flow stress for nickel has been reported to decrease sufficiently above 1100 K to allow for easy extrusion of metal parts[2]. When the zirconia powder compact densifies, graingrowth and pore coarsening occur simultaneously. The rate at which the zirconia densifies during hot pressing will thus decrease with increasing length of the coarsening step. Therefore, high densification rates should be expected for early application of the load, while lower densification rate are associated with late stress application. Once metal fully fills the pores in the ceramic, as shown in Fig 2, further densification is no longer possible and coarsening does not take place readily. When nickel is made to flow into the matrix early in the densification process, by appplication of a high stress, the pores in the ceramic are still small. Filling of these pores then leads to the formation of a layer through which further intrusion is more difficult than if the ceramic matrix had been allowed to coarsen first. The more rapid densification of the remaining matrix also closes the pores more rapidly. As a result, a decreasing extent of metal intrusion will occur by earlier load application during hot pressing.

B.Diffusion bonds

In the diffusion bonding at 1275 K, nickel was pressed onto zirconia sintered previously to some specific density. The pressure was kept well below the pressure used for intrusion bonding, at about 7 MN/m^2. Neither this bonding procedure nor the subsequent extended annealings led to any intermixing of significance, as should be expected for the low stress applied. These observations confirimed the explanation of the intrusion phenomenon as being due to nickel flow at high applied stress. The mechanical integrity of the diffusion bonds was in all cases quite poor, and often the junctions failed by spalling either immediately after the bonding or during the subsequent annealings. The further deterioration of the nickel /zirconia junction upon annealing in air is not surprising, considering the reactions that occur during this treatment.[3]

An interesting observation was that for prolonged annealings of the nickel/ zirconia bonded couples, at 1275 K, metal was lost from the interface, through open open pores in the ceramic. This is clearly shown in Fig. 3. Presumably, evaporation or surface migration is responsible for this phenomenon.

C.Indentation testing.

A rough estimate of the mechanical integrity or the thoughness of the different bond was obtained from Vickers micro-indentation tests. Results of such measurements are shown in Fig 4 for the hot pressed samples A and B that were also shown in Fig 1. Typically, for intrusion bonding the apparent hardness, which may be qualitatively associated with the local strength in the bond compared to the matrix, decreased with decreasing zirconia matrix density and increasing distance from the original Ni/zirconia interface. This is evident from the microhardness data shown in Fig 4 for the intrusion bonds A and C which were aslo shown in Fig. 1. Comparison of the results of such indentation tests for intrusion bonds and for diffusion bonds consistently showed that considerably higher mechanical integrity is associated with the intrusion bonding.

4. Conclusions

A graded ceramic metal junction can be produced by intrusion bonding of nickel to zirconia. The intrusion bond is obtained by hot pressing zirconia powder onto nickel sheet. The best bonds were achieved when high pressure was applied late in the heat treatment cycle, after the microstructure of the zirconia had been allowed to coarsen. The processing parameters controlling the quality of the intrusion bond include the magnitude of the applied pressure and the temperature and time at which it is applied . Diffusion bonding, either on dense or on porous zirconia, at low applied joining pressures around 1275K ,invariably led to poor bond characteristics which further degraded on prolonged annealing .

Acknowledgements

This work was supported by the Director, Divion of Meterials Sciences,Office of Basic Energy Research ,of the US Department of Energy under contract No DE-AC03-76SF00098.

References

1. K.Suganuma, T.Okamoto, M.Shimada, adn M.Koizuni.,"New Method for Bonding between Ceramics and Metals,"J. Amer. Ceram. Soc., C117-118 (1983)

2. K.Laue and H.Stenger,"Extrusion," Amer. Soc. Met., Metals Park, Ohio, 1981.

3. S.L.Shinde, I.E.Reimanis, and L.C.De Jonghe, "Evolution oif the Nickel/ Zirconia Interface," Ceram. Eng. Sci. Proc., Vol 7,1027- 31 (1986).

Table 1.

Sample	Porosity % Theoretical	Type of pores	intrusion distance in mm
Intrusion bonds			
A	8.9	open	0.76
B	2.3	closed	0.46
C	1.5	closed	0.05
Diffusion Bonds			
1	4.6	closed	
2	5.4		
3	6.2		
4	7.5	closed	
5	10.5		
6	11.2	open	
7	17.2	open	

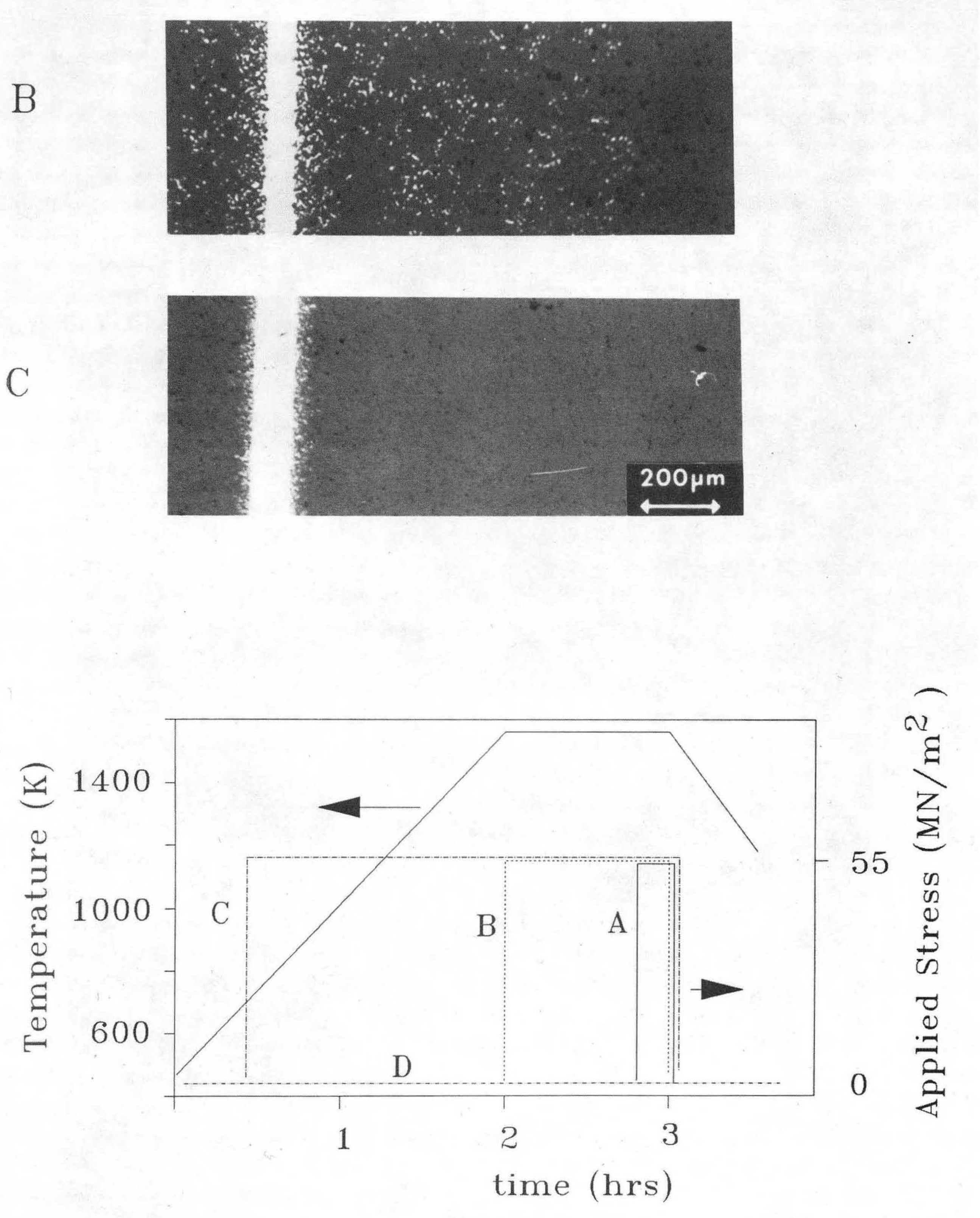
Figure Captions

Fig 1. Intrusion bonds of specimens A,B, C, D and their corresponding temperature, time, pressure history.

Fig 2.Close-up of the interface zone of an intrusion bond.

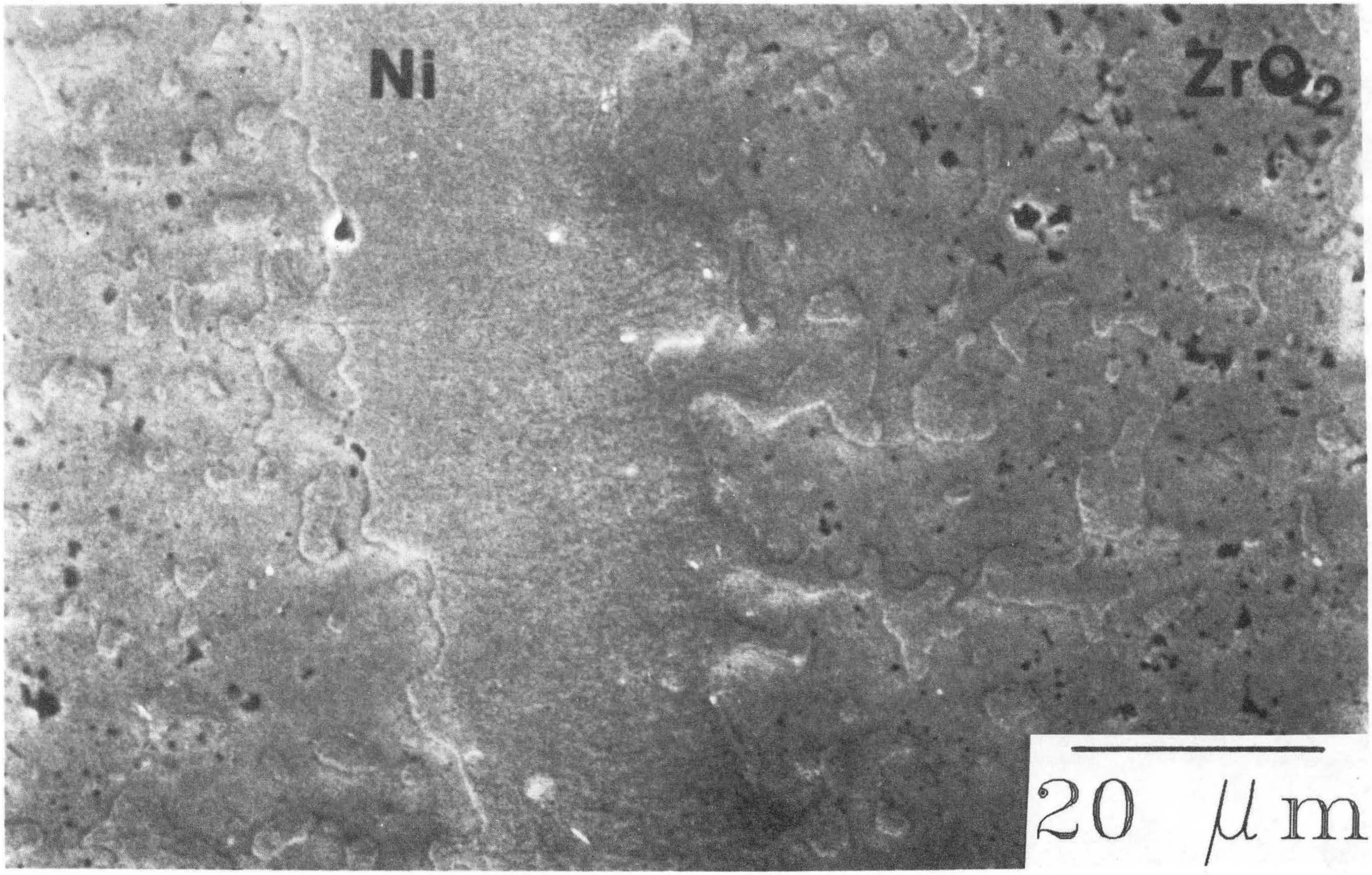
Fig 3. Pore formed in the nickel metal at the zirconia/ nickel junction, due to metal loss through the open pore network in the zirconia after a 1 hour anneal at 1275 K, in the diffusion bond of sample 7 (Table 1).

Fig 4. Vickers microhardness date for intrusion bonds A and C (see Fig 1)



XBB873-2196A

Fig. 1



XBB873-2206A

Fig. 2

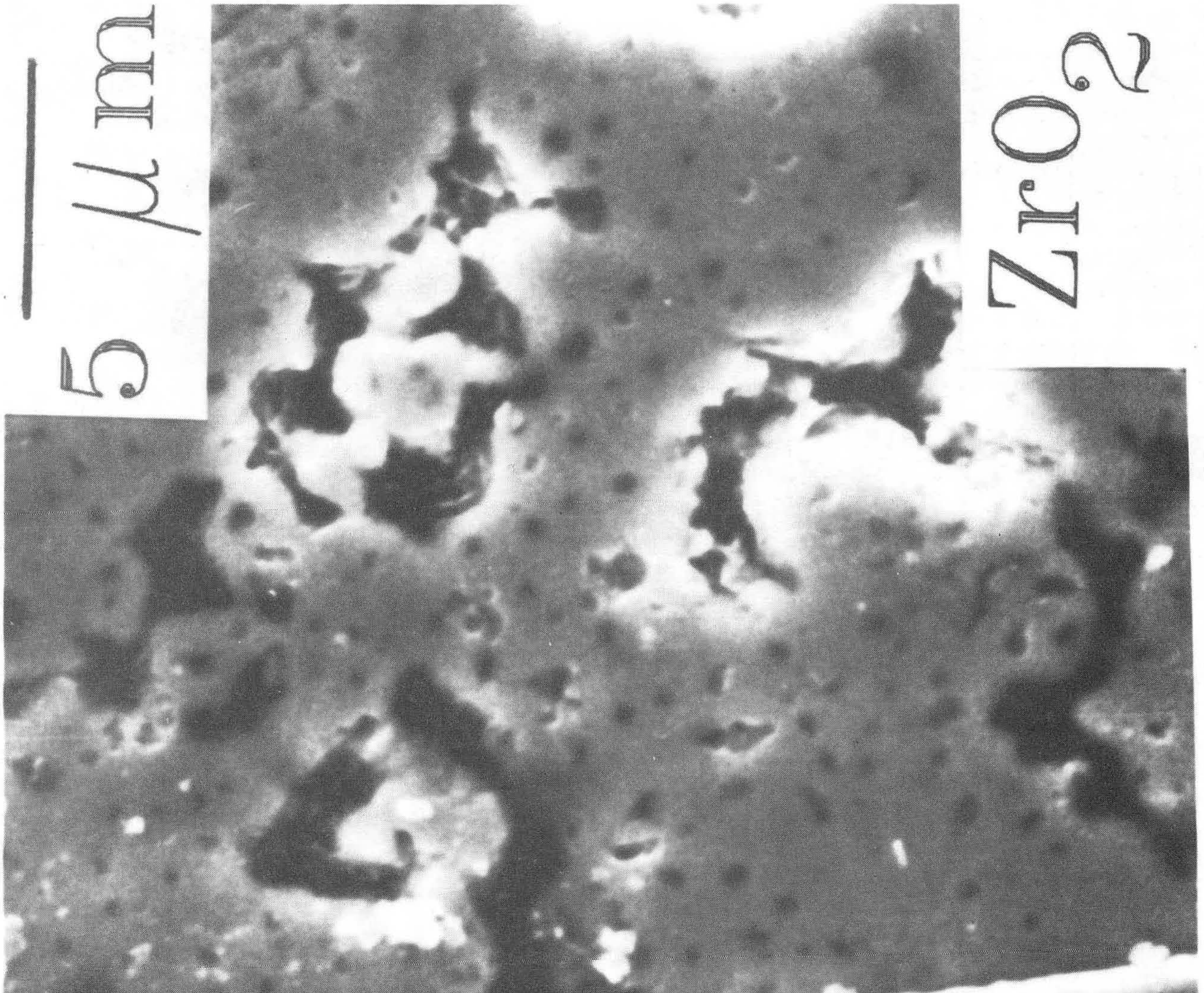
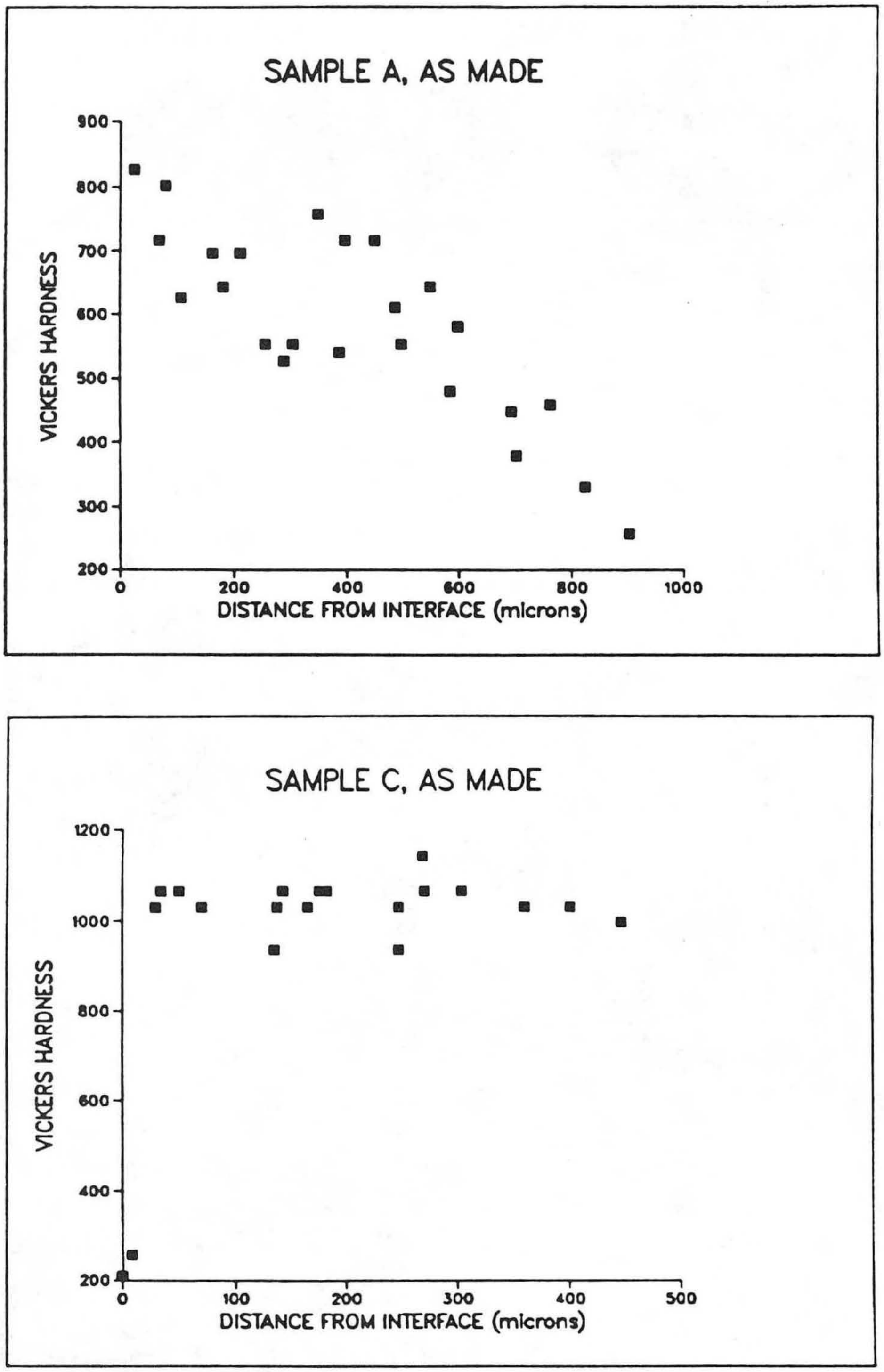


Fig. 3



XBL 873-1214 A

Fig. 4

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