ductors above some concentration depending perhaps on temperature (Wipf suggests 2.0% indium at about 3°K) one expects to find much larger tails on the magnetization curves of the more concentrated alloys than can be seen except at very high sensitivity. According to the theory of second-kind superconductors, the ratio of upper critical field H_{c2} to the thermodynamic critical field H_c for tin alloys would be

$$H_{c2}/H_c = K + 0.35\rho\ ,$$

where K is a constant and ρ is the resistivity in microhm-cm. Taking this ratio to be unity at 2.0% indium, one can calculate the separation between H_c and H_{c2}. If we neglect the difference between the lower critical field and H_c and assume H_c corresponds to the position of the rate-dependent peak in a rising field then we find that the calculated H_{c2} in each case (2.2%, 3.0%, 4.5%) is close to the end of the recovery of the normal signal. The recovery marks the end of the hysteretic tail, and thus provides a lower limit to the end of the tail if the hysteretic part is followed by a reversible region. The long shallow hysteretic tail is just visible in the part of the magnetization curve shown in Fig. 4 for the 4.5% alloy. It seems probable then that in our specimens of these alloys the tails due to second-kind superconductivity are present, but that they are very shallow indeed as compared with the height of the magnetization curve, and irreversible.

CONCLUSION

All the features of the behavior of our tin alloys (the same ones that were used by Wipf) in an alternating field can be related to hysteresis. To the extent that the appearance of hysteresis marks the onset of second-kind superconductivity, so (when the alternating field amplitude is very small) does the appearance of the peak in a steady field, as Wipf suggested. If, however, one believes that in a perfect specimen the transition would be reversible, then the criterion is not a very fundamental one, though in practice it may well work for specimens of attainable perfection as long as the amplitude is small enough. The rate-dependent peak phenomenon shows that the peak will reappear when the amplitude exceeds the separation of the steep parts of the magnetization curves, at any rate at low frequencies such as the 50 cps that we have used. As the sweep rate is raised there are signs of a slowing up of the maximum rate of change of flux in the transition of the 2.2% alloy, as shown both by the way in which the rate-dependent peak depends on sweep rate and by an increase in the hysteresis. This is the only alloy in which we have investigated this property. Whether it is a function of specimen quality, as we suppose the hysteresis and probably the shallowness of the tail on the magnetization curve to be, is a question we hope to answer by making carefully annealed single crystals of these alloys.

The Peak Effect in Substitutional and Interstitial Solid Solutions of High-Field Superconductors

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INTRODUCTION

An anomalous behavior of high-field superconductors is the "peak effect." The data have been displayed principally in either of two ways: (1) a peak in J_c, the critical current density vs H_t, the transverse magnetic field and (2) a dip in R, the resistance, vs H_t. The anomaly has been reported in cold-worked transition metal alloys[1] as well as in impure niobium[2] and in interstitial solid solutions.[3] In the latter case it has been associated with H_{c2}, the upper critical field for a "negative surface energy" superconductor (type II). The results, summarized here, show that the anomaly [the anomaly is to be distinguished from that observed in J_c vs H (longitudinal)[4]] is more readily observed in interstitial than in substitutional solid solutions of transition metals. In both materials, when the anomaly occurs, it takes place at H_{c2} as determined magnetically. The results sug-

[1] For earlier references on the peak effect, see T. G. Berlincourt and R. R. Hake, Phys. Rev. **131**, 140 (1963).
[2] S. H. Autler, E. S. Rosenblum and K. H. Gooen, Phys. Rev. Letters **9**, 489 (1962).
[3] W. DeSorbo, Bull. Am. Phys. Soc. **8**, 294 (1963).
[4] See for example, S. T. Sekula and R. W. Boom, Appl. Phys. Letters **2**, 102 (1963).

gest that the presence of solute or impurity atoms segregated at some structural irregularity such as dislocation is important for the appearance of the anomaly. Below H_{c2}, the resistivity decreases with cold-work in both types of solid solution. It is probably the type of resistance discussed by Kim, Hempstead, and Strnad.[5] Above H_{c2}, the resistance is probably normal, Ohmic resistance.

RESULTS AND DISCUSSION

The details of the measurements of critical current density J_c as a function of H_t, the transverse field, and of magnetic induction were similar to those described earlier.[6] The resistivity vs field curves (constant J) were evaluated from the voltage generated across a portion of the specimen measured to a sensitivity of approximately 0.1 μV. Details of the metal solution preparation have been given elsewhere.[6,7] The wire specimens were subjected to homogenization treatment at high temperatures by passing large currents in high vacuum (pressure less than 1×10^{-6} mm Hg).

The magnetization behavior of both substitutional[6] and interstitial[7] solid solutions and of Nb[8] has already been interpreted in terms of type II superconductors.[9,10] Structural irregularities and precipitates present in these superconductors are assumed to pin down flux filaments and act as free energy barriers. It is believed that the interaction of defects and flux filaments controls hysteresis of the type described by Bean[11,12] as well as critical transport currents.[5,13]

A comparison of the magnetization data (Fig. 1), obtained on a typical annealed interstitial (Nb + 0.7 at.% O) and substitutional (Nb + 3.0 at.% Ti) solid solution, with the data on threshold current density $J_c(H_t)$ vs field (Fig. 2) clearly shows three portions in the latter: (1) below H_{c1} a sharp decrease in J with H_t analogous to the behavior in a "soft" superconductor; (2) the "mixed state" region between H_{c1} and H_{c2} where the critical current increases with cold-work; and (3) a region above H_{c2}.

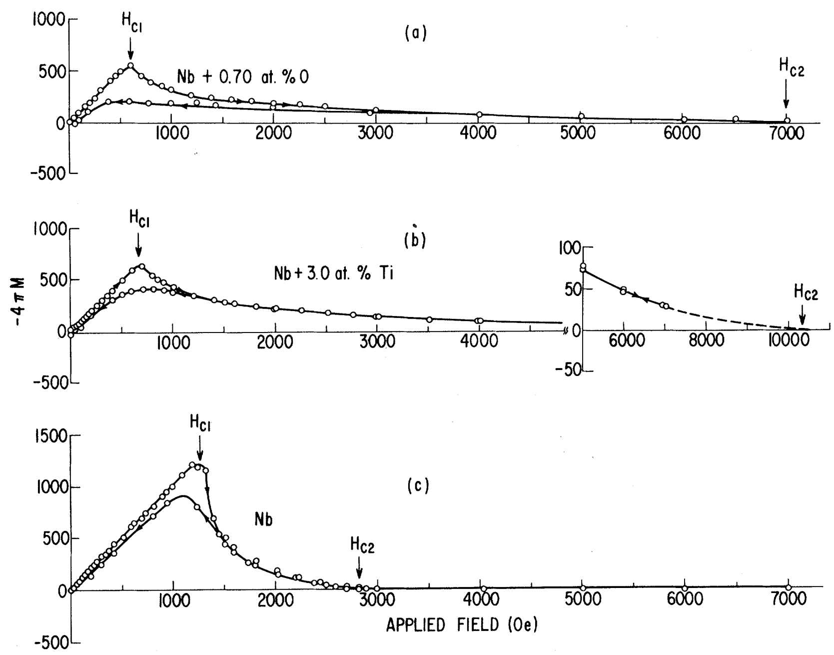

Fig. 1. The magnetization of (a) interstitial solid solution, niobium +0.70 at.% oxygen homogenized 1 h at 1100°C; (b) substitutional solid solution, niobium + 3.0 at.% titanium homogenized 4 h at 1700°C; (c) niobium electron-beam melted, outgassed, and annealed. $R_{300°K}/R_{10°K} \simeq 500$. $T = 4.2$°K.

In the "mixed state" of the annealed wires of Nb + 3.0 at.% Ti and Nb + 0.7 at.% O (Fig. 2), the increase in J_c culminating in a peak occurs only in the interstitial solution. When these wires are given similar histories of cold-work (uniformly compressed to a ribbon), the peak is evident in both solid solutions only when the field is perpendicular to the wide side (w.s.) of the ribbon ($H \perp$ w.s.). If the field is

[5] Y. B. Kim, C. F. Hempstead, and A. R. Strnad, Phys. Rev. **131**, 2486 (1963).
[6] W. DeSorbo, Phys. Rev. **130**, 2177 (1963).
[7] W. DeSorbo, Phys. Rev. **132**, 107 (1963).
[8] T. F. Stromberg and C. A. Swenson, Phys. Rev. Letters **9**, 370 (1962).
[9] A. A. Abrikosov, Zh. Eksperim. i Teor. Fiz. **32**, 1442 (1957) [English transl.: Soviet Phys.—JETP **5**, 1174 (1957)].
[10] B. B. Goodman, IBM J. Res. Develop. **6**, 63 (1962).
[11] C. P. Bean, Phys. Rev. Letters **8**, 250 (1962).
[12] J. Silcox and R. W. Rollins, Appl. Phys. Letters **2**, 231 (1963).
[13] J. Friedel, P. G. DeGennes, and J. Matricon, Appl. Phys. Letters **2**, 119 (1963).

parallel to this side ($H \| $w.s.), no peak is evident. Whenever the peak appears, it does so at a field coinciding with H_{c2} determined from the magnetization data (compare Figs. 1 and 2). This observation is consistent with that made earlier by Hake, Berlincourt, and Leslie[14] who reported the peak to occur at

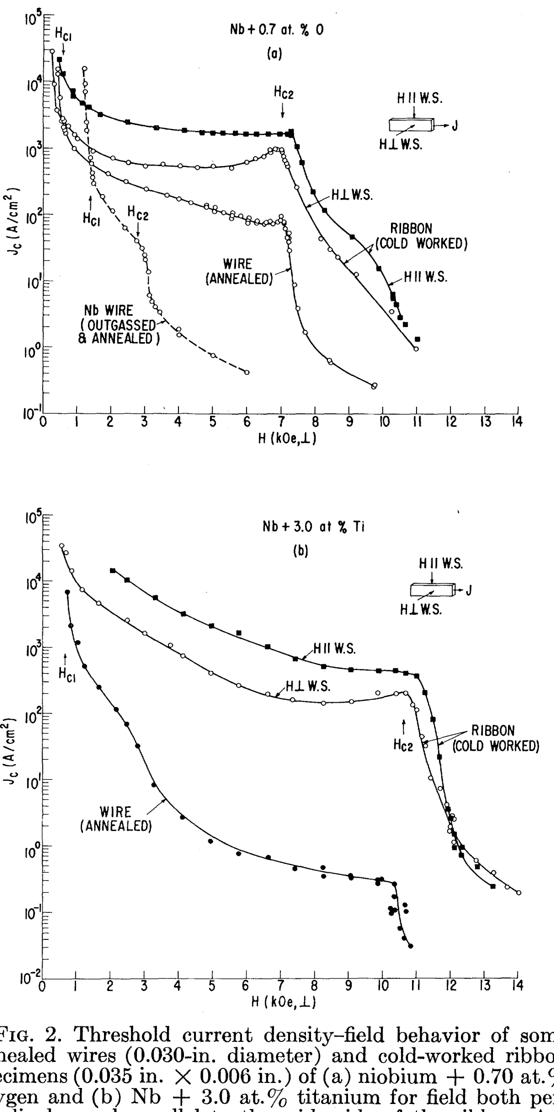

Fig. 2. Threshold current density–field behavior of some annealed wires (0.030-in. diameter) and cold-worked ribbon specimens (0.035 in. × 0.006 in.) of (a) niobium + 0.70 at.% oxygen and (b) Nb + 3.0 at.% titanium for field both perpendicular and parallel to the wide side of the ribbon. Annealed and outgassed Nb wire (0.030-in. diameter) is also shown for comparison. $T = 4.2°$K.

a field just below the "upper" resistive critical field, $H_r(J = 10 \text{ A/cm}^2)$. A comparison of Figs. 1 and 2(b) shows that this arbitrarily defined field is only slightly larger than H_{c2} in cold-worked substitutional solutions. However, in the interstitial solutions [Fig. 2(a)], the discrepancy between the two may be appreciable. In both systems, this resistive critical field is structure sensitive.

The resistance vs field data are summarized in Fig. 3 for some ribbon specimens where the field is perpendicular to the current flow and to the wide side ($H \perp$ w.s.). R is derived from the voltage as a function of H_t (J constant), and R_n from the voltage observed when the material becomes completely normal. The dip in the curves, following an initial rise, is related to the peak in the $J_c(H_t)$ curve, and, similarly, appears in the interstitial solutions both before and after the cold-work but in the substitutional only after cold-worked. These minima also appear at H_{c2}.

When the field is parallel to the wide side of the ribbon ($H \|$w.s.), the resistance minimum is still present occuring at H_{c2}, but is less pronounced. With this field orientation, the resistance below H_{c2} for a given J is reduced appreciably.

In polycrystalline niobium, outgassed and annealed ($T \simeq 1800$°C, vacuum less than 1×10^{-7} mm Hg, $R_{300°\text{K}}/R_{10°\text{K}} \simeq 500$) where the total measured interstitial content is less than 5 ppm, no peak is evident [Fig. 2(a)] at the precision of this work. This is true even when cold-worked. These results suggest that a solute or impurity atom is probably necessary for the appearance of the peak. The effect of cold-work on the Nb–Ti and Nb–O alloys suggests that segregation of these impurity atoms to dislocations may be of importance. The peak effect seems to be more readily apparent in interstitial rather than substitutional solid solutions. The anomaly has been seen in niobium containing less than 200 ppm of oxygen. Interstitial atoms have far greater mobility than substitutional ones, and hence can more easily segregate to structural irregularities such as dislocations. Stiegler *et al.*[15] have recently shown that the presence of relatively small amounts of interstitial impurities in niobium (probably less than 150 ppm) has a strong influence on annealing structures of the metal. Dislocations were reported to act as sinks for interstitials present as interstitial atmospheres or precipitates. These results indicate that small amounts of interstitial impurities may have been responsible for the appearance of the anomaly reported earlier in niobium.[2,16,17]

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[14] R. R. Hake, T. G. Berlincourt, and D. H. Leslie, Bull. Am. Phys. Soc. **7**, 474 (1962).

[15] J. O. Stiegler, C. K. H. Dubose, R. E. Reed, Sr., and C. J. McHargue, Acta Met. **11**, 851 (1963).

[16] T. G. Berlincourt, Phys. Rev. **114**, 969 (1959).

[17] M. A. R. LeBlanc and W. A. Little, in *Proceedings of the Seventh International Conference on Low-Temperature Physics*, edited by G. M. Graham and A. C. Hollis-Hallet (University of Toronto Press, Toronto, 1961), p. 362.

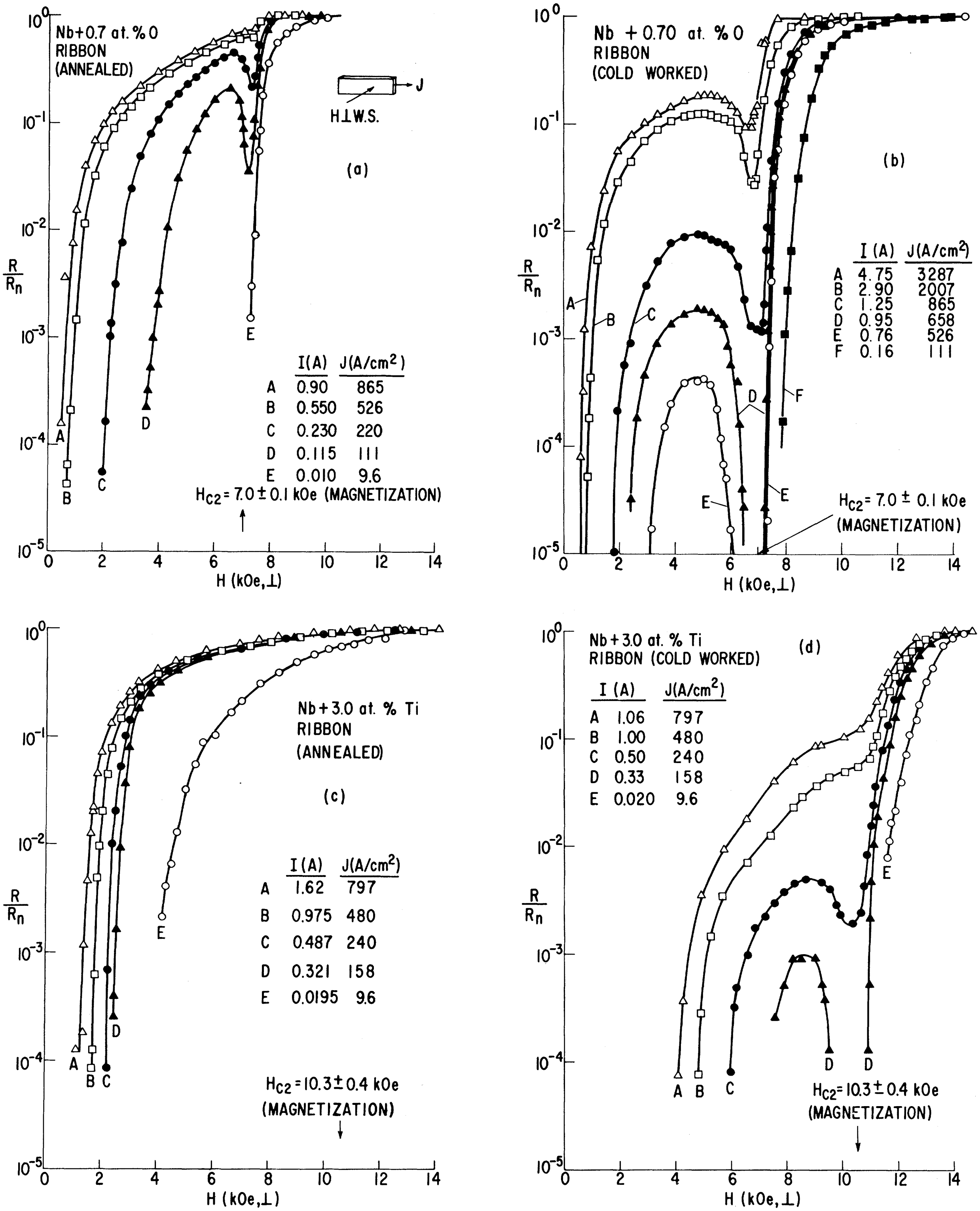

FIG. 3. Resistance-field data for various current densities for both annealed and cold-worked ribbon specimens (0.035 in. × 0.006 in.) of (a) niobium + 0.70 at.% oxygen and (b) niobium + 3.0 at.% titanium. Field perpendicular to wide side of the specimen. $T = 4.2°K$.

Some additional experiments were done to vary the degree of interstitial segregation to dislocations. Rapid quenching of Nb + 0.7 at.% oxygen from an homogenization temperature (e.g. $\simeq$ 1100°C) tends to minimize the effect. Cold-working such a quenched sample enhances the effect. A low-temperature anneal (3 h at 170°C) decreases the resistivity and strongly decreases the peak. Increasing the solute concentration above the solubility limit also masks the peak effect. These results suggest that the peak effect is strongest at some intermediate stage of the process of segregation and precipitation at dislocations.

Hauser and Treuting[18] have suggested that anisotropy in defect structure is probably responsible for the minimum ("valley effect") in the $J_c(H)$ curve but reported no evidence for the presence of a second phase. Preliminary optical microscopy studies on some of the specimens reported here revealed precipitate particles only in cold-worked specimens (ribbon) occuring at a high density in certain areas, presumably regions of greater dislocation density.

Although the present experiments outline the dependence of the peak effect on various structural features, a satisfactory theory of this effect has not yet been developed. Presumably, in the mixed state, the "resistance" may be of the kind discussed by Kim, Hempstead, and Strnad.[5]

[18] J. J. Hauser and R. G. Treuting, J. Phys. Chem. Solids **24**, 371 (1963).

ACKNOWLEDGMENTS

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Discussion 11

MENDELSSOHN: Listening to Dr. DeSorbo I think I have found a possible explanation of the peak effect. It seems significant that the peak does not occur at the place where you can attain the highest current density. The peak occurs in a transverse field; but a higher current can be put through the specimen in a longitudinal field. Perhaps in the transverse case the current follows more or less a straight path through a sponge network of the specimen. But when you approach H_{c2} where superconductivity would be quenched, the current could take bending paths through the network. These bent paths would then be acted on by a more or less longitudinal field in which case the current is allowed to have a higher value than when the paths were straight and transverse to the field.

WARREN DESORBO, *General Electric Research Laboratory:* Your explanation is a plausible one. Unfortunately we have not investigated these samples in a longitudinal field to make the comparison you mention.

MENDELSSOHN: Isn't there a higher current density for a given field when the direction is changed?

DESORBO: There is a higher current density when the field direction is changed from perpendicular to parallel (to the wide side of the ribbon), where the peak disappears in the latter orientation.

GORTER: Earlier, at this conference, I suggested that this peak effect might be due to a sort of matching between the layer structure and the irregularities in the metal. Several other people have also mentioned this. This point of view would quite well agree with the comments of Dr. DeSorbo, as well as with the remarks made by Dr. Mendelssohn. If we have a not too high field in a superconductor of the second kind, the layer structure (or if you wish the flux line structure) takes a more or less favorable position with respect to the irregularities which are in the metal. Near the upper critical field, the order parameter ψ^2 goes to zero at certain places. Then you automatically go over into a more loose structure which has the character of a sponge. This structure is finally the last possibility for superconduction because, if you continue to increase the field, ψ^2 becomes zero at too many places, and the sample becomes normal. That currents parallel to the field can be larger than those perpendicular to the field is evident because the Lorentz force is absent in one case and present in the other.

S. H. AUTLER, *Massachusetts Institute of Technology:* A few comments on our observations on the occurrence of this peak or dip. The samples that Dr. Rosenblum was talking about were well-annealed Nb samples in which most of the dislocations had probably been removed. Further evidence that this was so was the fact that the magnetization curves taken on the same samples were fairly close to being reversible, indicating that perhaps on the macroscopic scale these were fairly perfect specimens. I might say in corroboration of Dr. DeSorbo's results that we tended to see this resistance dip with less frequency on the purer samples, although we have seen it for a sample with a resistance ratio as high as 500. There is no exact correlation between resistance ratio and the occurrence of the dip; we have some reason to suspect perhaps surface effects are involved.

DESORBO: I was going to ask you if you observed the dip after etching a specimen.

AUTLER: We haven't done a very systematic job. We have found that, on some occasions, etching a sample made the dip appear where it hadn't been present before; on other occasions it made it disappear when it had been present before.