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# MULTIPLE ION IMPLANTATION EFFECTS ON HARDNESS AND FATIGUE PROPERTIES OF Fe-13Cr-15Ni ALLOYS

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## Abstract

Eight complex alloys based on the composition Fe-13Cr-15Ni-2Mo-2Mn-0.2Ti-0.8Si-0.06C were implanted simultaneously with 400 keV boron and 550 keV nitrogen, and investigated for microhardness changes and bending fatigue life. The dual implantation was found to decrease the fatigue life of all eight alloys although the implantation increased near-surface hardness of all eight alloys. This result was in contrast to the significant improvements found in the fatigue life of four B, N implanted simple Fe-13Cr-15Ni alloys. It was determined that the implantation suppressed surface slip-band formation, the usual crack initiation site, but in the complex alloys, this suppression promoted a shift to grain boundary cracking. A similar phenomenon was also observed when the simple Fe-13Cr-15Ni alloys were simultaneously implanted with boron, nitrogen and carbon wherein fatigue life decreased, and again, grain boundary cracks were observed. To test the hypothesis that ion implantation made the overall surface more fatigue-resistant but led to a shift to grain-boundary cracking, single crystal specimens of the ternary Fe-15Cr-15Ni were also implanted with boron and nitrogen ions. The fatigue life decreased for the single-crystal specimens also, due to concentration of applied stress along fewer slip bands as compared to the control single crystal specimens where applied stress was relieved by slip band formation over the entire gauge region. Fatigue life improvement by ion implantation at intermediate strain levels has a sensitive dependence on grain size as well as extent of slip band suppression.

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## Introduction

Ion implantation of metals has previously been shown to be an effective technique in many cases for improving fatigue properties. Implanting the metal surface with energetic ions delays and suppresses the formation of slip bands (the preferred initiation site for fatigue cracks) at the surface, thereby increasing the overall fatigue life [1,2]. An earlier study by Lee and Mansur showed that simultaneous dual implantation of four Fe-13Cr-15Ni model alloys with boron and nitrogen increased hardness and improved fatigue life to a greater extent than did single implantation with either ion species [3]. Their results indicated that multiple ion implantation could be an effective technique for significantly improving fatigue properties.

In the present study, the hardness and fatigue properties of eight complex engineering Fe-13Cr-15Ni alloys implanted simultaneously with boron and nitrogen were investigated. These alloys are based on the composition Fe-13Cr-15Ni-2Mo-2Mn-0.2Ti-0.8Si-0.06C. In an earlier paper, we showed that the hardness increased but the fatigue life of these alloys decreased after the dual implantation [4]. This decrease was attributed to a shift to grain boundary cracking from slip-band crack formation. To further explore this hypothesis, single-crystal specimens of an Fe-15Cr-15Ni alloy were implanted with boron and nitrogen ions and tested for fatigue life. In the present paper, the single-crystal results are compared with the results obtained using polycrystalline specimens, and possible mechanisms of fatigue-crack initiation in ion-implanted metals are examined.

## Experimental Procedure

The compositions of the alloys, designated as E-1 through E-8 are given in table 1. The single-crystal specimens were grown using the Czochralski method and had a composition close to Fe-15Cr-15Ni as determined by chemical analysis. The single-crystal samples were oriented using x-ray back reflection and then machined using electric-discharge erosion. The longitudinal sample axis was oriented along [100]. All specimens were implanted simultaneously with 400 keV boron and 550 keV nitrogen to a dose of $2.3 \times 10^{16}$ ions/cm$^2$ for each ion. The implantations were carried out at the triple ion irradiation facility in the Metals and Ceramics Division at Oak Ridge National Laboratory using the 400 kV and 2.5 MV Van de Graaff accelerators, respectively, for boron and nitrogen [5]. The peak in the depth profile of the implanted species was determined to be approximately 0.5 μm for all of the implantations as estimated by TRIM90 calculations [6].

Miniature bending-fatigue specimens with the configuration shown in figure 1 were used for the fatigue tests. In the case of the implanted specimens, only the hourglass-shaped gauge region was implanted. Special holders were designed for irradiating the specimens in the accelerator chamber. Separate holders were required to implant the edges and surfaces; each specimen was subjected to four irradiations, one for each surface and edge. The specimens were electrochemically polished using a perchloric acid-ethanol electrolyte and then annealed for 15 minutes at 1150°C in vacuum prior to implantation.

Fatigue tests were performed using a specially designed test machine developed at Auburn University. The specimen is a cantilever beam with a bending load applied at the free end. Specimens were tested at constant amplitude at a flexing frequency of 8.33 Hertz (500 cycles per minute) with a total deflection of 4.8 mm at the point of loading corresponding to a strain range of 0.21% in the necked region with a zero mean stress representing an intermediate strain level. All specimens were tested at a single value of deflection for comparison purposes. The tests were terminated when the load experienced by the specimen decayed to 80 % of the original load, as detected by the load cell. Further details concerning the special test machine may be obtained from reference [7]. Optical microscopy was used to study the crack initiation and slip-band morphology on the surface of the specimens after fatigue testing.

## Results

The results of the fatigue tests using the eight E-series alloys as well as the single-crystal specimens are shown in figure 2. The control specimen results are the average of 2 to 4 specimens while the implanted-specimen results were obtained using one specimen per alloy. It can be seen that the fatigue life of the eight complex alloys decreased after dual implantation, ranging from a 15% decrease for alloys E-1 (base) and E-6 (50% less Si) to 45% for alloy E-7 (no Si). On the other hand, the fatigue life of the single-crystal specimen also decreased after implantation by about 8% for one specimen and 26% for the other. Knoop microhardness measurements were carried out on a Leitz Metalloplan optical microscope with a video camera attachment and television screen to image and accurately measure the diagonal of the Knoop diamond indents. Knoop hardness results are also shown in figure 2 for all alloys. A load of 5 gms was used for the Knoop tests. A lower load would have made it difficult to accurately estimate the indent diagonal length while a higher load would have included effects from the unimplanted deeper regions in the hardness measurement. It can be seen in figure 2 that the surface hardness increased for all the alloys after implantation, by up to a factor of two, indicating a strengthening of the implanted layer.

Optical microscopy revealed a definite suppression of slip bands in all the implanted specimens as compared to the control specimens. Figure 3 shows typical micrographs of the fatigue cracked surface in the gauge region of the unimplanted and implanted specimens for the polycrystalline specimens as well as for the single crystal specimens. In the control specimens, slip bands were visible throughout the entire gauge region. The control specimens showed one-to-three major cracks and a number of microcracks. In the polycrystalline control specimens, the extent of slip-band formation varied from grain to grain depending on the orientation with respect to the loading axis. Since the single-crystal specimens were oriented with the longitudinal axis along the [100] direction, two sets of slip bands were observed in the gauge region. These slip-bands were perpendicular to each other and both were oriented 45° with respect to the [100] crystallographic direction. These slip-bands correspond to two $\{111\}<110>$ systems, the preferred slip system for fcc metals, each of which is active during the tension and compression cycles, respectively. Slip bands were observed to be present over the entire gauge region of the control single-crystal specimens. In case of the implanted specimens, typically one major crack and a few microcracks were visible. As seen in figure 3, slip bands were confined to the immediate vicinity of the major crack where the applied strain had the greatest value. The area covered by slip bands was much lower as compared to the control specimens.

Fatigue cracks can initiate at slip bands, twins, grain boundaries, and at precipitate particles. For the single-crystal specimens, both control and implanted, the cracks always initiated at slip bands. Twin bands were also observed in the implanted single-crystal specimens although fatigue cracks did not initiate at twin bands. Interrupted tests were conducted on control polycrystalline specimens with the test stopped at various intervals and the specimen examined in the optical microscope in order to examine crack-initiation sites. For the control polycrystalline specimens, cracks were found to initiate mainly at slip bands. A number of grain-boundary cracks were also formed in the later stages of the test. The final major crack was found to originate from cracks initiated at slip bands. Interrupted tests were not performed on the implanted specimens. However, observations of failed implanted polycrystalline specimens using the optical microscope revealed a number of grain boundary cracks in the gauge section as shown in figure 4. Fatigue cracks in implanted single-crystal specimens always initiated at intense slip bands.

## Discussion

In earlier studies, fatigue cracks have been shown to initiate at persistent slip bands (PSBs) [8]. The PSBs, which penetrate the surface of the specimen, have intrusion-extrusion topographies, and fatigue cracks were shown to initiate at the edge where the PSB emerges from the surface under some of the intrusions [8]. Ion implantation causes the formation of a thin, hard layer with an increased yield strength on the surface causing a reduction in the surface plastic strain, thereby preventing slip bands in the bulk from penetrating to the surface. Thus, slip bands could penetrate the implanted layer only in a small region of maximum applied stress but were effectively prevented from appearing at the surface in the rest of the gauge region by the implanted layer. This suppression of slip band formation at the surface with the consequent delay and suppression of fatigue crack initiation has been determined to be responsible for improvements in the fatigue life [9,10].

The microstructure of the implanted layer is of significance for achieving an understanding of the mechanisms responsible for the prevention of slip-band penetration in the implanted layer. Radiation damage is an important factor, and the ion implantation process creates point defects that can form clusters and Frank loops which hinder dislocation motion and prevent slip-band penetration. Self ($Fe^{++}$) ion implantation of Fe-13Cr-15Ni alloys, however, caused no significant changes in the fatigue life indicating that in this case, radiation damage may not be a dominant factor [4]. Self implantation did cause an increase in surface hardness as has also been shown in other studies [9,11].

The formation of precipitate particles can hinder dislocation motion, thereby preventing slip bands from penetrating the implanted layer. Transmission electron microscopy of the B, N implanted layer, however, did not reveal the presence of precipitates [4]. The failure to observe precipitate particles in the earlier study was attributed to the obscuring effects of overlapping strain fields due to a high concentration of point defects and to the modest implant doses employed. The results suggest that any precipitates that may have formed were too small to be detected. Earlier studies of nitrogen-implanted Fe-Ni-Cr alloys [12] and 304 type stainless steel [13] did, however, reveal the presence of nitride clusters.

It is clear from figure 3 that slip-band formation was obviously suppressed at the surface. The reduction in fatigue life due to the dual implantation can be explained in terms of a transformation to grain boundary cracking from slip-band crack initiation. Grain-boundary cracks were observed even in the unimplanted E-alloy specimens after fatigue tests, and many grain-boundary cracks were evident in the gauge region of the implanted specimens. Slip bands in the implanted specimens were suppressed from penetrating the implanted layer to such an extent that the applied stress was relieved by cracking of favorably oriented grain boundaries where the adjacent grains had a high degree of misorientation and the grain boundary was oriented approximately 90° relative to the load axis.

In our previous study, four simple Fe-13Cr-15Ni alloys (see table 2 for compositions) were either singly implanted with boron and nitrogen, simultaneously implanted with boron and nitrogen, or simultaneously implanted with boron, nitrogen, and carbon [3,4]. The fatigue-specimen configuration as well as implant and test conditions were identical to those used in this study. The energy for carbon implantation was 400 keV. The fatigue test results are shown in figure 5. Single ion implantation caused at most modest improvements in the fatigue life whereas dual implantation yielded significant improvements (up to 250% for alloy B-6). Interestingly, triple ion implantation caused a decrease in fatigue life. Optical microcopy revealed the presence of grain boundary cracks only in the triple-implanted specimens [4]. Since the E-alloys are inherently stronger than the B-alloys due to solute and precipitate strengthening, dual implantation was sufficient to cause the transformation to grain- boundary cracking whereas in the B-alloys only triple implantation caused sufficient strengthening for such a transformation.

Such an effect is not possible in the single crystal specimens due to the absence of grain boundaries. However, while slip was suppressed to a great extent in the BN implanted single-crystal specimens, some slip bands did manage to penetrate the implanted layer to the surface. Thus, further applied stress was concentrated in these slip bands leading to crack initiation earlier than that for the control specimens. In the implanted B alloys, fatigue life improved due to two possible reasons: Firstly, only some of the grains were oriented favorably for forming slip bands and consequently, cracks. Secondly, slip bands that did form were probably blocked by grain boundaries, delaying crack initiation and propagation. Only in the triple implanted specimens were the slip bands suppressed to such an extent to cause grain boundary cracking.

It is clear that there is an optimum level of surface strengthening by ion implantation for suppressing slip band penetration and improving fatigue life. Further strengthening causes grain boundaries, which can be viewed as pre-existing flaws,to come into play. This result was also shown in a dose-dependence study by Hohmuth et. al. [14] where polycrystalline nickel was implanted with boron at different doses. These authors found an optimum dose for the maximum fatigue-life improvement while higher doses caused a reduction in fatigue life. This observation was again attributed to the promotion of grain-boundary crack formation.

The fatigue test results presented here are limited to a single strain level and to a single dose for each ion. However, there is a clear dependence on grain size as evinced by the single crystal results. The single crystal specimens had stress concentrated along some slip bands leading to an earlier crack initiation at these bands. The more effective strengthening of the E-alloys preferentially initiated cracks at grain boundaries again decreasing fatigue life. Thus, improving strain-controlled fatigue properties of metal alloys at intermediate strain levels by ion implantation has a sensitive dependence on grain size as well as extent of suppression of slip at the surface.

## Conclusions

The primary conclusions resulting from the present investigation are:

(1) Simultaneous implantation of boron and nitrogen in eight Fe-13Cr-15Ni-2Mo-2Mn-0.2Ti-0.8Si-0.06C based complex alloys caused a significant improvement in hardness. However, the fatigue life of all eight alloys decreased.

(2) The decrease in the fatigue life of the implanted E-alloys was attributed to a transformation to grain-boundary cracking from slip-band crack initiation, which is the operative failure mechanism in the unimplanted alloys.

(3) Supporting evidence was obtained from an earlier study of multiple-ion implantation of four

simple Fe-13Cr-15Ni alloys. Dual implantation of these alloys with B and N significantly improved the fatigue life, but triple ion implantation with B, N, and C decreased fatigue life. Only the triple implanted specimens showed grain-boundary cracks.

(4) Single-crystal Fe-15Cr-15Ni specimens were used in order to eliminate grain-boundary effects. The single-crystal specimens were also implanted with boron and nitrogen ions. The fatigue life of single-crystal specimens also decreased after implantation. This was attributed to concentration of stress along fewer slip bands leading to earlier crack initiation as compared to the control specimen.

(5) The present results indicate that there is an optimum level of strengthening by ion implantation for the prevention of slip-band penetration of the implanted layer and corresponding improvement of fatigue life. Any further strengthening causes the stress to be relieved by grain boundary failure which then becomes the fatigue crack-initiation site leading to an overall lower fatigue life.

(6) Improving strain-controlled fatigue properties of alloys by ion implantation has a sensitive dependence on grain size as well as on extent of suppression of slip at the surface.

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Table 1

E-series alloy compositions in weight percent

| ALLOY | Fe | Cr | Ni | Mo | Ti | Si | Mn | C | P | ASTM Grain Size |
|---|---|---|---|---|---|---|---|---|---|---|
| E-1 | Bal. | 13.14 | 15.10 | 1.98 | 0.19 | 0.82 | 2.13 | 0.059 | 0.002 | 5.0 |
| E-2 | Bal. | 13.41 | 14.97 | 0.01 | 0.19 | 0.80 | 2.16 | 0.055 | 0.003 | 4.7 |
| E-3 | Bal. | 13.19 | 15.07 | 1.97 | 0.17 | 0.82 | <0.01 | 0.053 | 0.002 | 6.0 |
| E-4 | Bal. | 13.21 | 15.11 | 1.98 | <0.01 | 0.83 | 2.13 | 0.040 | 0.002 | 3.5 |
| E-5 | Bal. | 13.18 | 15.15 | 1.98 | 0.17 | 0.83 | 2.14 | 0.009 | 0.002 | 5.1 |
| E-6 | Bal. | 13.24 | 15.07 | 1.97 | 0.17 | 0.37 | 2.14 | 0.047 | 0.002 | 6.0 |
| E-7 | Bal. | 13.33 | 15.05 | 1.97 | 0.17 | 0.01 | 2.16 | 0.044 | 0.002 | 5.6 |
| E-8 | Bal. | 13.15 | 15.12 | 1.98 | 0.17 | 0.84 | 2.14 | 0.054 | 0.044 | 5.5 |
| Single Crystal | Bal. | 15.0 | 15.0 | - | - | - | - | - | - | - |

Note: Trace elements Nb, B, N, O, S, V, Cu, and Al less than 0.01 wt %.

Table 2

Composition of B-series alloys in weight percent

| ALLOY | Fe | Cr | Ni | Mo | Ti | Si | C | P |
|---|---|---|---|---|---|---|---|---|
| B-1 | Bal. | 13.79 | 14.96 | - | - | - | - | - |
| B-5 | Bal. | 13.68 | 14.94 | - | 0.17 | - | 0.040 | - |
| B-6 | Bal. | 13.36 | 15.04 | 1.97 | 0.19 | - | 0.056 | - |
| B-12 | Bal. | 13.58 | 15.15 | - | 0.17 | 0.83 | 0.044 | 0.049 |

## Figure Captions

1. Miniature bending fatigue specimen configuration used in this study.

2. Fatigue test and microhardness results for the unimplanted and implanted complex E-alloys and the single crystal specimens, showing the increases in hardness (below) and decreases in fatigue life (above) for the implanted specimens (SC indicates a single crystal specimen; two specimens, SC7 and SC8 were implanted and tested).

3. Typical optical micrographs of the gauge regions of unimplanted and implanted E-alloys and single crystal specimens showing the suppression of slip bands. Note the symmetric pattern of slip bands in two <111>{110}systems active in the tension and compression cycles respectively in the single crystal specimen.

4. Typical grain boundary cracks observed in the implanted specimens after fatigue testing which probably lead to earlier failure of the specimen.

5. Fatigue test results for the simple B-alloys showing that dual implantation significantly improves fatigue life whereas triple implantation decreases fatigue life due to the shift to grain boundary cracking (from Ref [3]).

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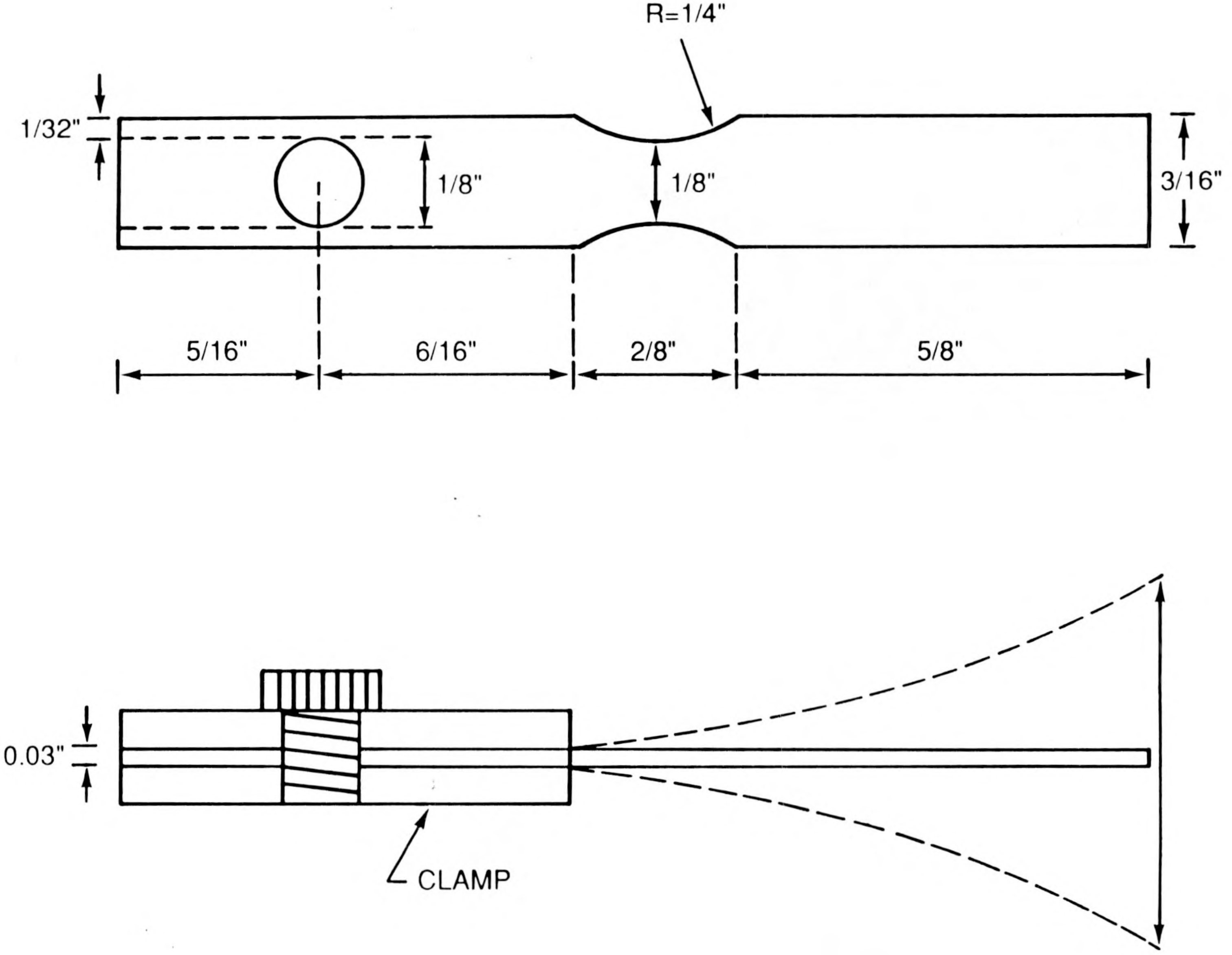

Fatigue Specimen Configuration

Figure 1 (Rao et. al.)

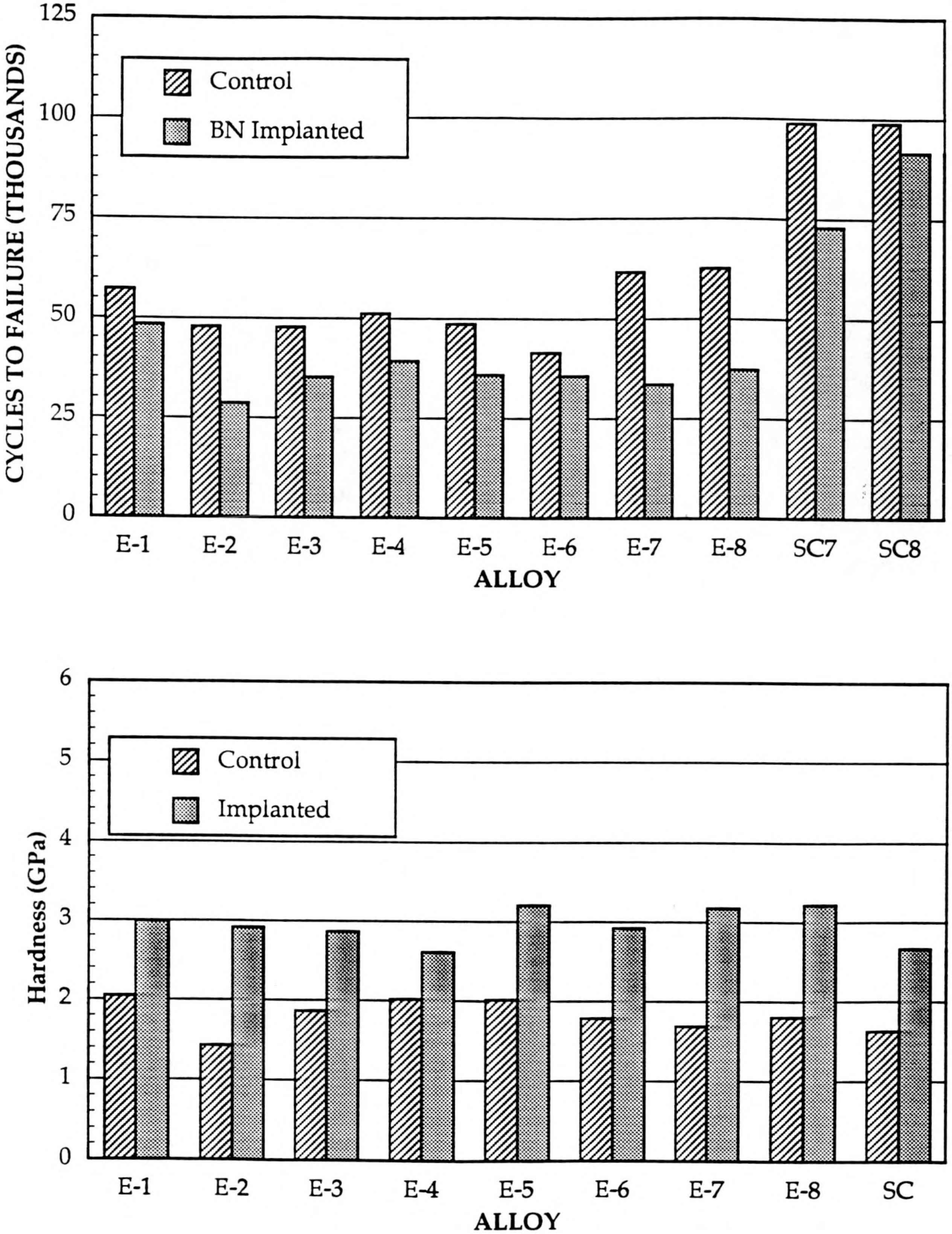

Figure 2

Figure 2 (Rao et. al.)

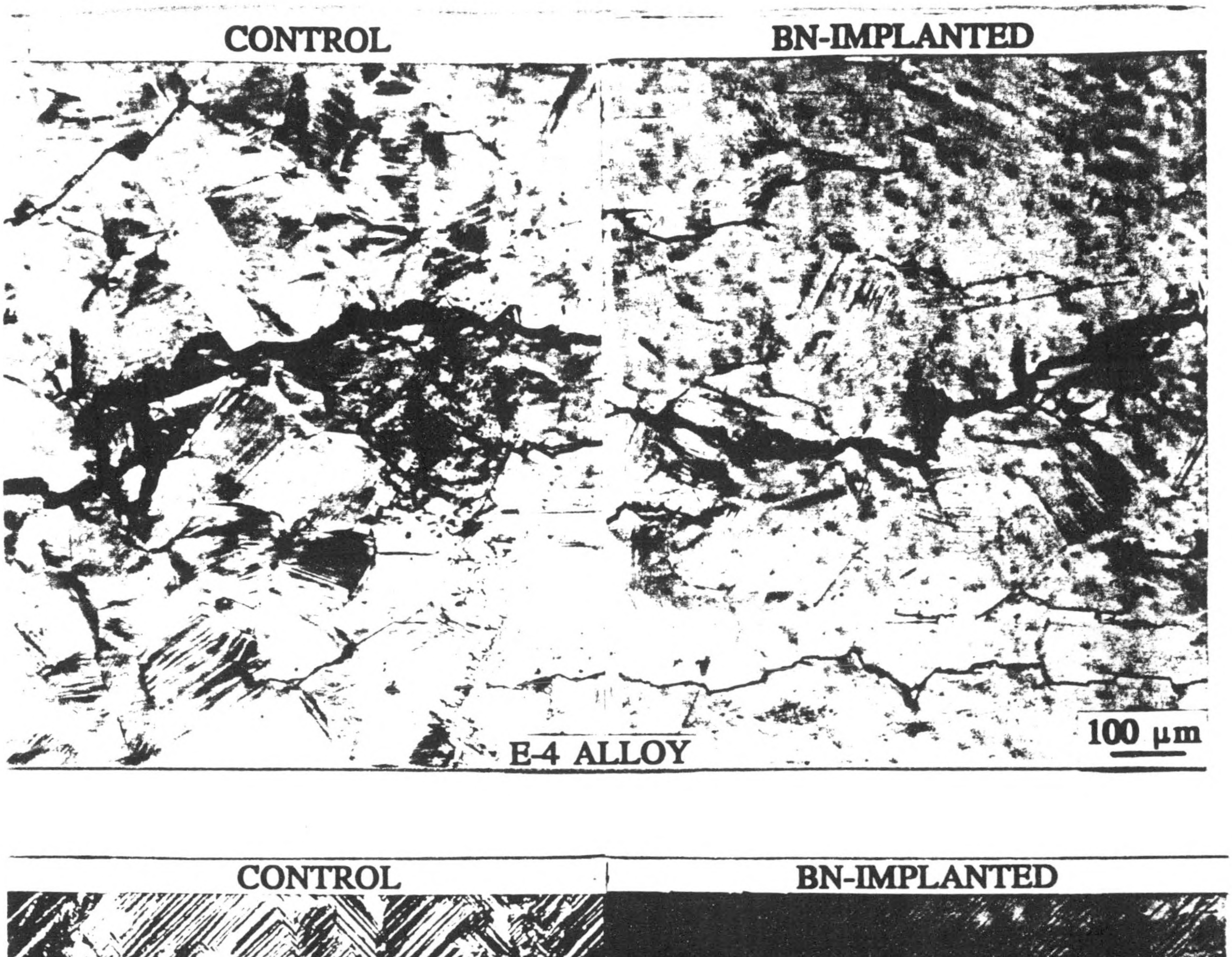

Figure 3 (Ram et. al.)

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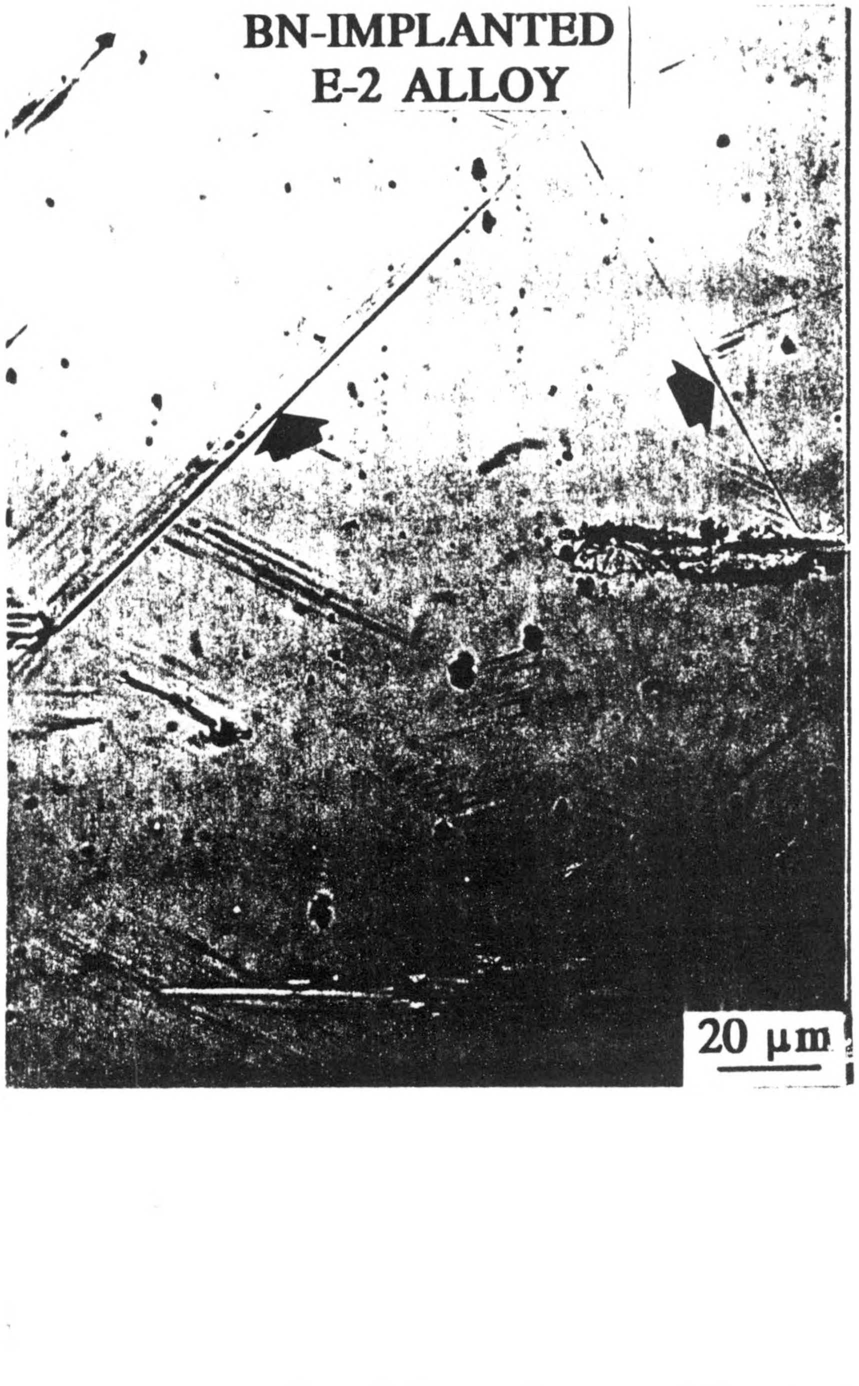

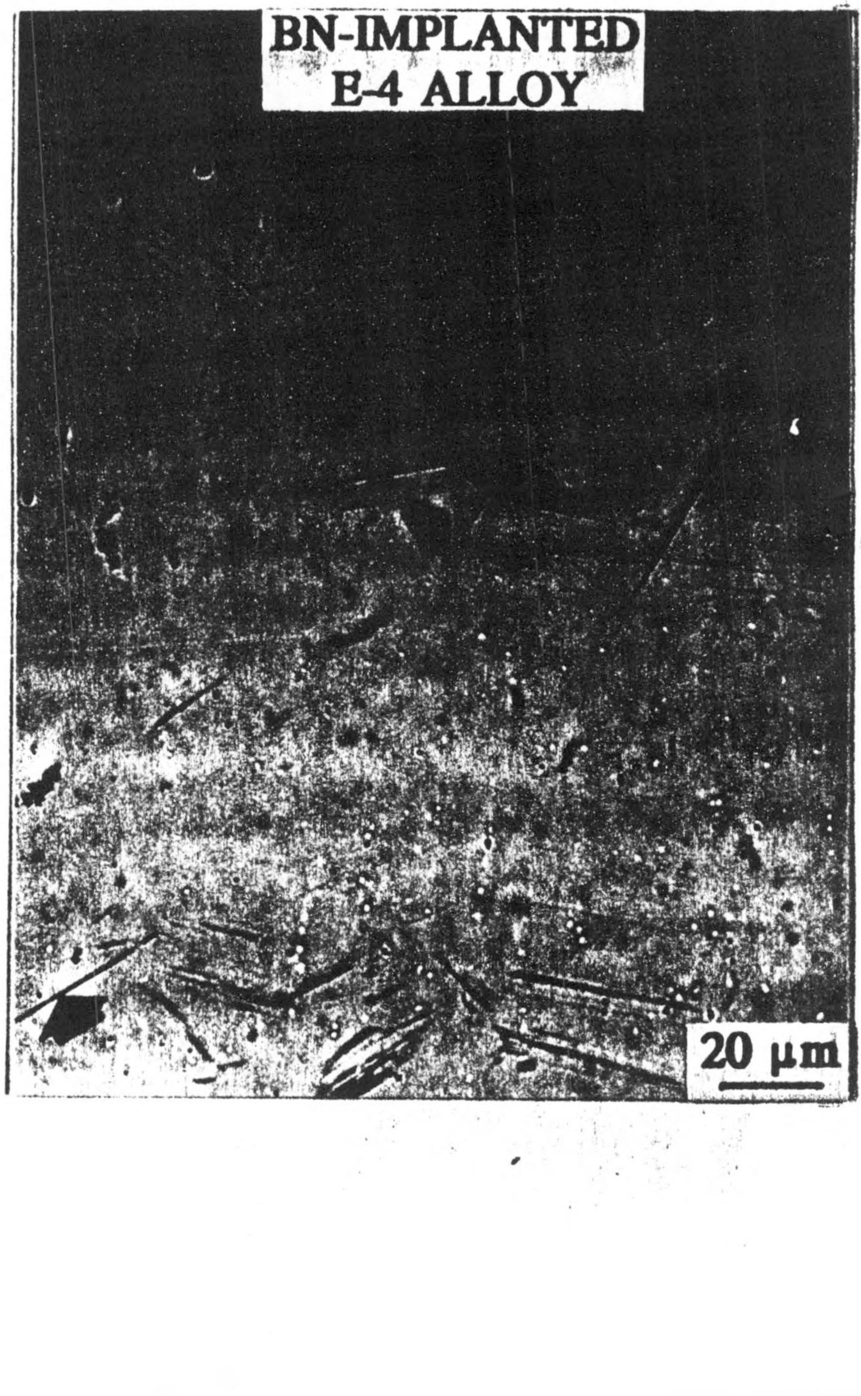

Figure 4 (Rao et al.)

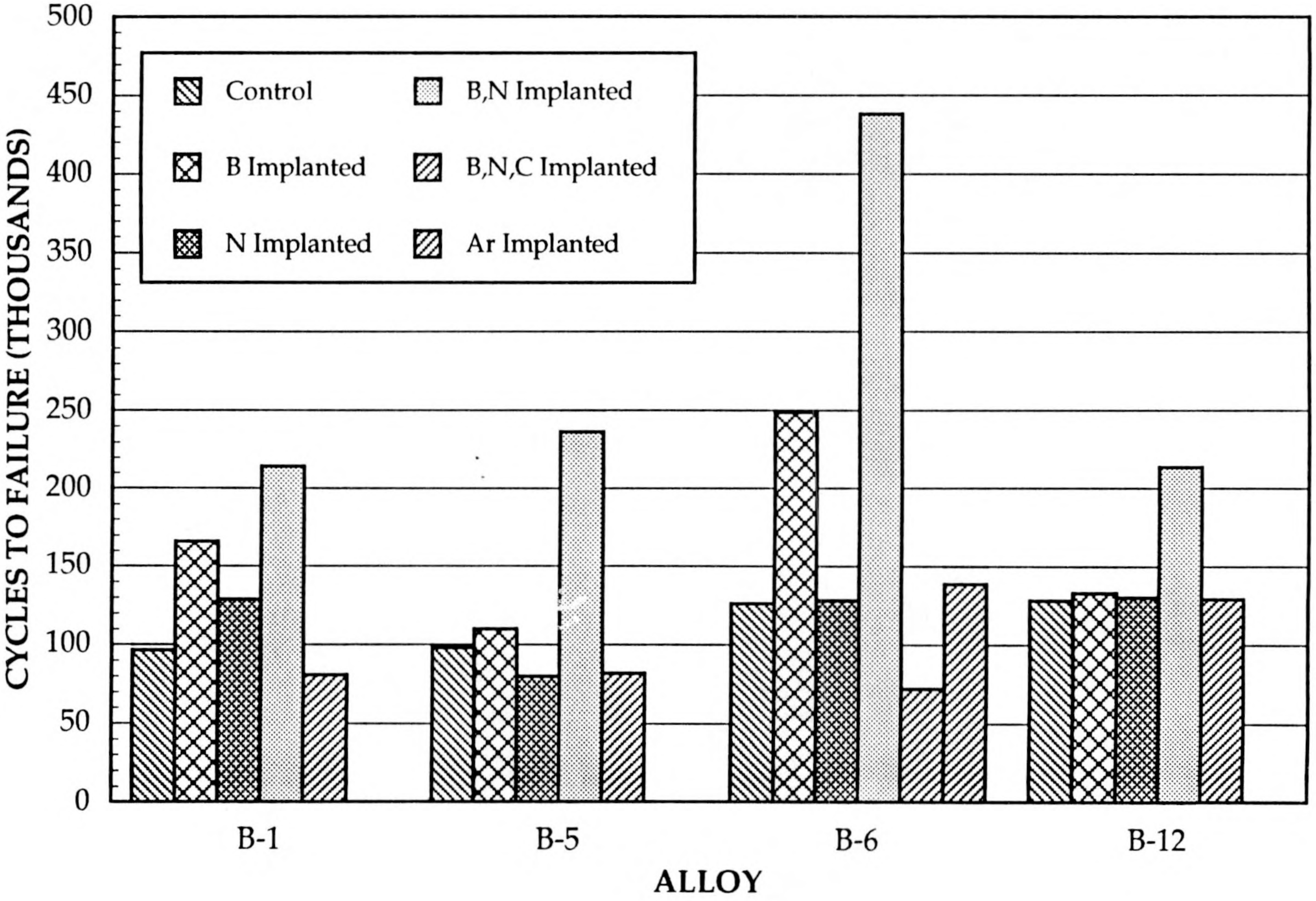

Figure 5 (Rau et. al.)