The Cross-Section Techniques for Preparing TEM Specimens of Heavy Ion Irradiated HT-9 Ferritic Steel

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THE CROSS-SECTION TECHNIQUE FOR PREPARING TEM SPECIMENS OF HEAVY ION IRRADIATED FERRITIC STEELS

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Summary

A new technique that involves iron plating on heavy ion irradiated ferritic steels (i.e., HT-9 and 2 1/4 Cr-1 Mo) to prepare transmission electron microscopy (TEM) specimens for cross-sectioned analysis has been developed. The procedures that relate to a successful experiment are described in detail. The same technique can also be applied to other ferritic steels and α-iron with minor modifications.

Introduction

In heavy ion irradiation experiments, the damaged region is limited by the injection distance of the ions into the material. Usually, the damage region is contained in the first few microns from the surface. Figure 1 is a typical damage level versus depth curve of 14 MeV Ni ions injected into HT-9. It is clearly seen that the damage level varies by factors of four or more in a relatively short distance. Figure 1 also shows the deposited position of injected ions which is between the peak damage and the end of damage.

By using the conventional back-thinning technique (CBT), only a certain depth of damage level can be examined at one time (i.e., the shaded CBT region in Fig. 1). This method produces some error due to the uncertainty of the pre-removed depth. It also has the disadvantage that the effect of the injected ions cannot be examined. Therefore, it is essential to develop the cross-section technique in order to examine the whole damage region at one time.

Experimental Procedures

The experimental procedures are divided as follows:

A. Pre-irradiation Sample Preparation

B. Post-irradiation Sample Preparation

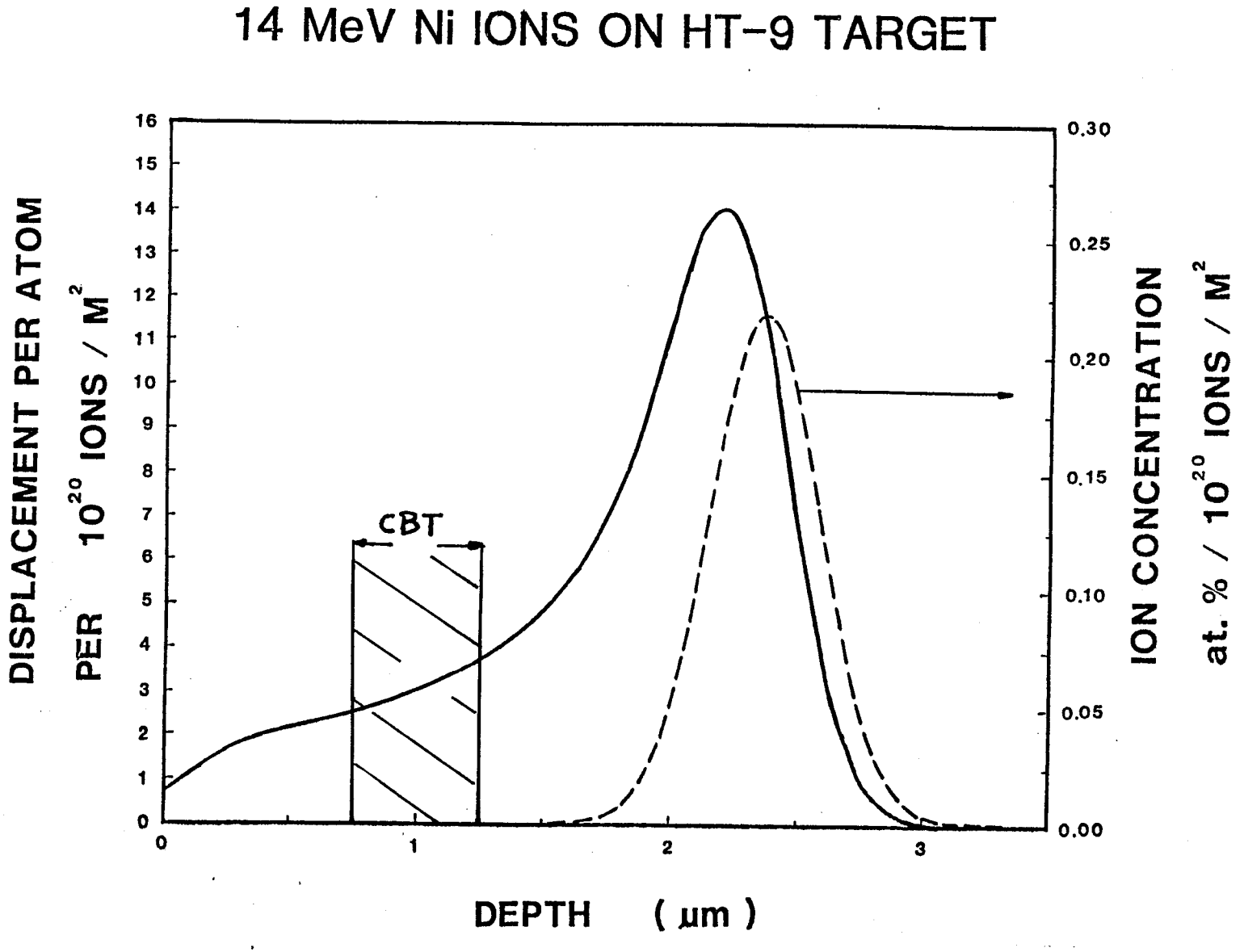

Fig. 1. The dpa and ion deposition vs. depth curves of 14 MeV Ni ion on HT-9 target.

B.1. Electroplating

B.2. Slicing and Cutting

B.3. Electropolishing

B.4. Ion Milling

It is convenient to consider each step individually.

The following procedures are used for HT-9 steel, which only need minor changes for 2 1/4 Cr-1 Mo steel and will be described as needed. The experimental procedures used for 2 1/4 Cr-1 Mo steel were first developed by R.L. Sindelar[1].

A. Pre-Irradiation Sample Preparation

To avoid introducing gas atoms into the specimen (e.g., during electropolishing), each specimen that will be used in an irradiation study is mechanically polished. The specimens have dimensions of 10 mm x 5 mm x 0.76 mm. After the mechanical polishing, the specimens are stored in plastic capsules filled with methyl alcohol until the time for irradiation.

B. Post-Irradiation Sample Preparation

B.1. Electroplating

1. Remove the irradiated specimen from the freezer several hours before the actual plating and allow the specimen to warm up to room temperature.
2. Fix the plating solution:
   - 200 gm $FeCl_2 \cdot 4H_2O$
   - 75 gm NaCl
   - 75 gm $CaCl_2$
   - 850 ml H_2O

3. Filter the plating solution with No. 1 filter paper to remove the dark brown oxides and obtain a clean green solution. It usually takes about three hours to accomplish this step.

4. Heat the plating solution (on a heating plate) to 94°C.

5. During the whole plating process, the gas bubbler should be used to keep the whole solution at the same temperature as the thermocouple readings. Figure 2 shows a schematic of the sample holder and gas bubbler.[2]

   Note: (i) The nitrogen gas used for the gas bubbler should be the high purity, dry grade.

   (ii) The bubbling speed is important in order to obtain an even and adherent plating.

6. At the same time of heating the plating solution, the irradiated specimen is slightly mechanically polished with 0.05 μm Al_2O_3 powder and mounted on the specimen holder. Figure 3 shows the schematic of the position of the specimen holder in the plating bath.

   Note: The specimen surfaces should face the anodes and the distance from the specimen surface to the anode should be no farther than a few centimeters.

7. Fix the pickling solution:

   - 2 gm thiourea (NH_2CSNH_2)
   - 15 ml H_2SO_4
   - 80 ml H_2O

   Note: The pickling solution works better for HT-9 at warmer conditions. Therefore, the time difference between actually mixing the solution to pickling the specimen should be minimized.

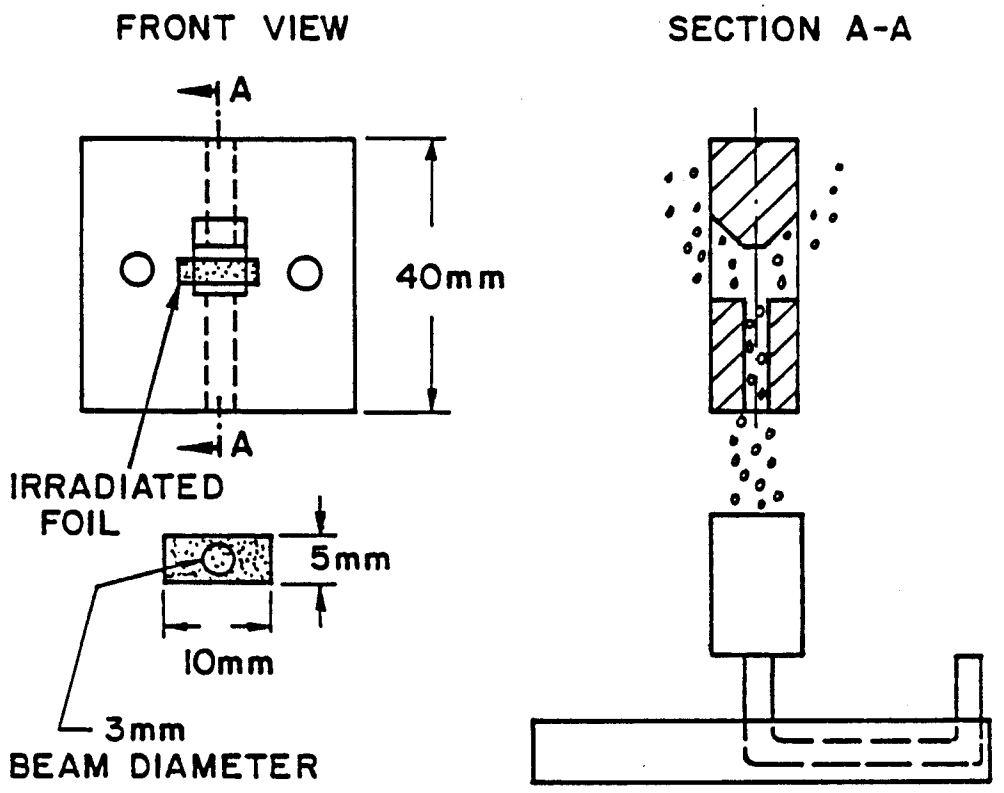

Fig. 2. The schematic of sample holder and gas bubbler.

SCHEMATIC OF THE WHOLE PLATING ASSEMBLY

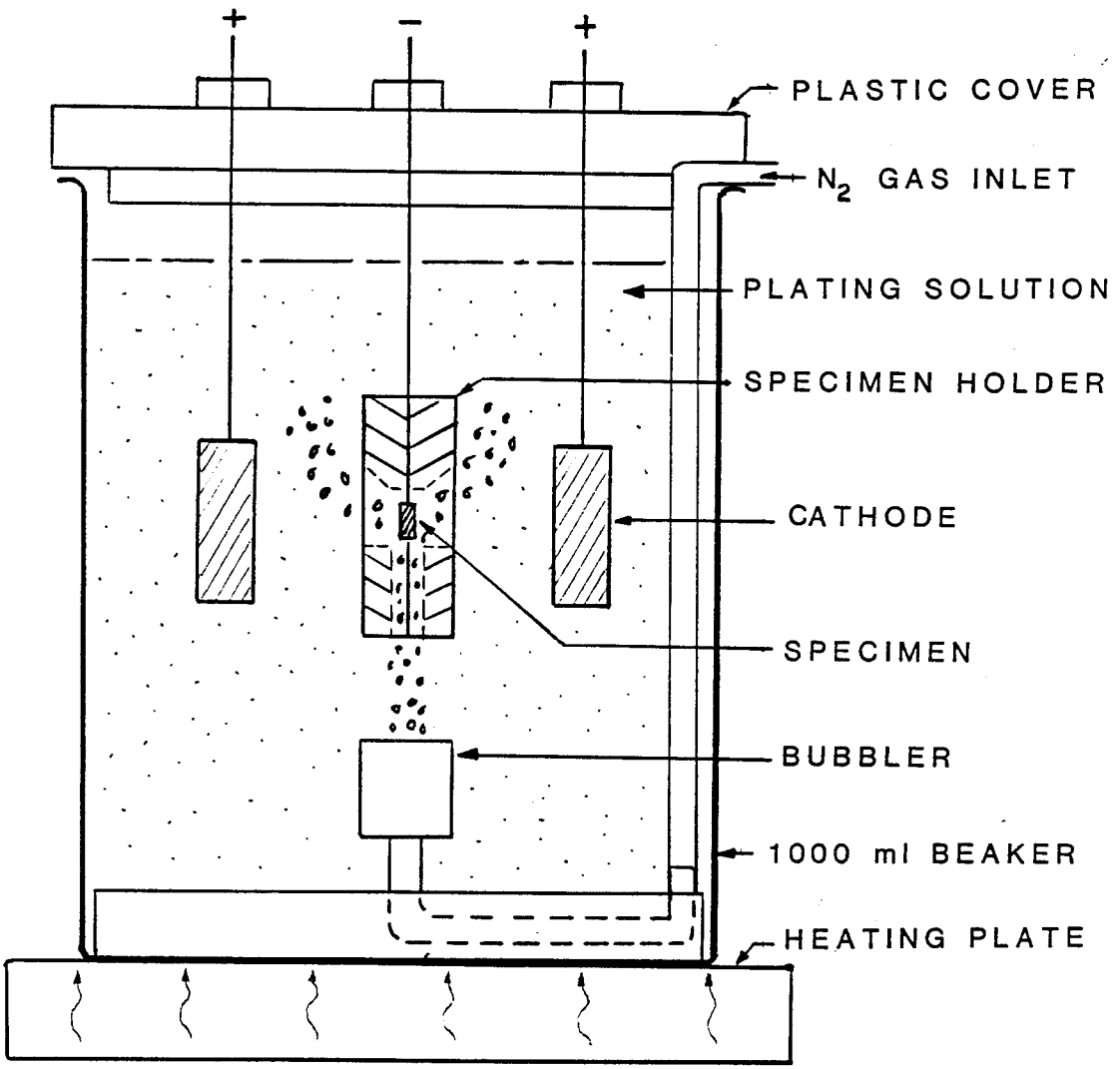

Fig. 3. The schematic of the whole plating assembly.

8. Pickle the specimen, which is mounted on the specimen holder, for 3 1/2 minutes.

9. Quickly wash the specimen and the holder with distilled water spray and immediately transfer the whole assembly into the 94°C plating solution.

10. Strike at 94°C, 100 mA for 7 seconds.

11. Start plating the specimen at 70 mA and set the automatic temperature control at 88°C.

12. It usually takes about 20 hours to plate a thickness of thicker than 3 mm.

    Note: The pickling process loosens the surface oxides of the specimen and the striking process actually removes these oxides. The surface that is removed by these two processes is about 50 nm which is negligible compared to the 2,000 to 3,000 nm range of the incident particle.

B.2. Slicing and Cutting

1. After electroplating, the specimen is mounted in a container filled with epoxy to form a rectangular block.

2. Slice in the direction vertical to the irradiated surface with a low speed diamond saw.

    Note: The thickness of each slice will determine the final TEM specimen, therefore, it is important to keep the thickness below 0.25 mm, especially for magnetic materials (e.g., HT-9 and 2 1/4 Cr-1 Mo steels).

3. There are usually 6 to 7 slices that contain an irradiated region and each of them has the potential to become a successful TEM specimen.

4. If the interface bond is good, each slice can be punched into a 3 mm disc with a mechanical puncher.

5. If the bond is not strong enough to hold for punching, then the slice is cut into a 3 mm disc by using a rotary cutter with a 3 mm inside diameter hollow tool bit.

B.3. Electropolishing

1. Etch the 3 mm disc specimen in a picral solution (1 gm picric acid, 5 ml HCl, and 100 ml methyl alcohol) for 20 seconds.

   Note: This process can make the interface between HT-9 and plated iron clearly show up which is necessary for painting lacquer to prepolish the specimen.

2. Paint lacquer over the entire specimen except for a 50 μm wide stripe along the irradiated interface. Figure 4 illustrates this step.

3. Electropolish to form a dish along the interface which will insure that the final hole will form along the irradiated interface.

4. Remove the lacquer cover with acetone.

5. Accomplish the final perforation by electropolishing in the same polishing solution.

   Note: (i) The polishing solution: (for HT-9)

   10% $HClO_4$ + 90% ethyl alcohol.

   (ii) Polishing condition:

   a. - 20°C, 60 mA for prepolishing

   b. - 20°C, 80 mA for final perforation.

   (iii) The prepolishing time depends on the thickness of the specimen and the unpainted area, therefore, it is essential to conduct experiments to determine the best condition. For

SCHEMATIC OF PAINTING LACQUER FOR PREPOLISHI

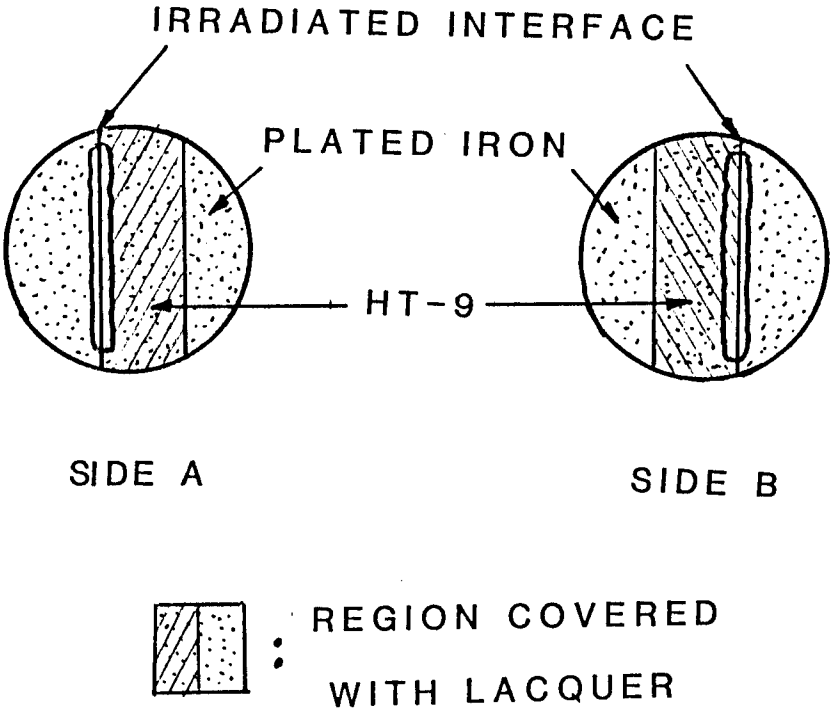

Fig. 4. The schematic of painting lacquer over a specimen for prepolishing.

HT-9, the prepolishing time is about 1 1/4 seconds per μm thickness of the specimen.

(iv) The polishing solution and condition: (for 2 1/4 Cr-1 Mo steel)

- 5% $HClO_4$ + 95% ethyl alcohol
- 60 V, 0°C, 90 mA

6. If the hole was perforated right on the irradiated interface, the sample preparation procedures are finished.
7. If the hole was not perforated along the interface, then the specimen needs to be thinned further by an ion mill.

B.4. Ion Milling

1. In most cases, the perforation will not form right on the irradiated interface and it is necessary to extend the thin area by ion milling.
2. Measure the distance between the edge of the hole to the interface by an optical microscope to determine the milling condition.
3. If the distance is large (e.g., greater than 20 μm), then the specimen needs to be milled at high voltage and high angle (e.g., 6 kV and 15° injection angle). If the distance is short, the voltage and the angle can be reduced.
4. The ion milling process is performed at liquid nitrogen temperature to avoid surface damage.
5. Use low angles and low voltages (e.g., 2.5 kV and 11.5°) for final thinning.

Note: From previous experience, if the hole is in the HT-9 portion, one can always obtain some thin area along the irradiated interface. But if the hole is in the plated iron portion, it is difficult to get good thin area.

Results

Figure 5 shows a sequence of steps of an irradiated cross-section specimen. The top shows the plated sample in the rectangular block, and next the slice and the 3 mm disc after punching. Figure 6 shows an optical micrograph of iron plated HT-9 specimen which was irradiated at 500°C and the peak damage level of 200 dpa. The photograph clearly indicates the interfaces between iron and HT-9 and the irradiated side is shown by the arrow.

Figure 7 is a low magnification TEM micrograph showing the extent of the electron-transparent region along the irradiated boundary of a cross-sectioned HT-9 specimen. The specimen was irradiated at 500°C and 40 dpa with 100 appm He preimplanted. Figure 8 is a high magnification TEM micrograph showing the detailed microstructures of the HT-9 specimen that was irradiated at 500°C and 100 dpa peak damage level. The detailed discussion of the microstructural evolution of HT-9 under heavy ion irradiation has been described elsewhere.[3]

Conclusion

The whole process from getting the sample after irradiation to producing a successful TEM specimen usually takes about one week to complete. A considerable amount of patience and care is required for a successful experiment.

The same technique can also be used for other ferritic steels and α-Fe except for some minor changes. For example, the pickling time and condition may vary with the stabilization of surface oxides, the prepolishing time may vary with the chemical composition of the material, etc.

This technique works quite well for both HT-9 and 2 1/4 Cr-1 Mo steels and we see no reason why it should not work for other steels. One major disadvantage of this technique is the plated iron is strongly magnetic which will affect the specimen examination in a TEM. However, since the HT-9 and 2

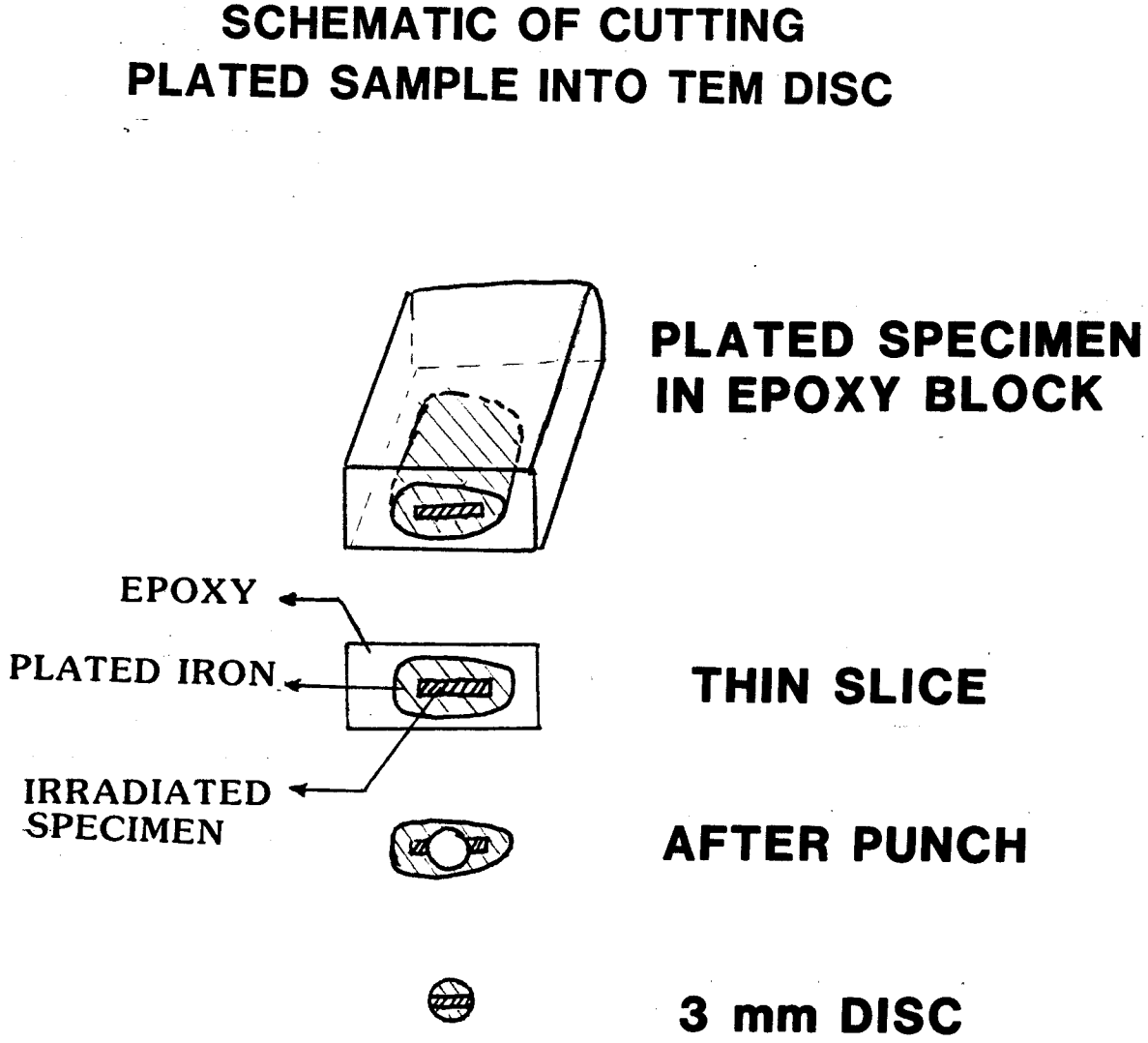

Fig. 5. The cross-sectioned HT-9 specimens appear immediately after being sliced and punched.

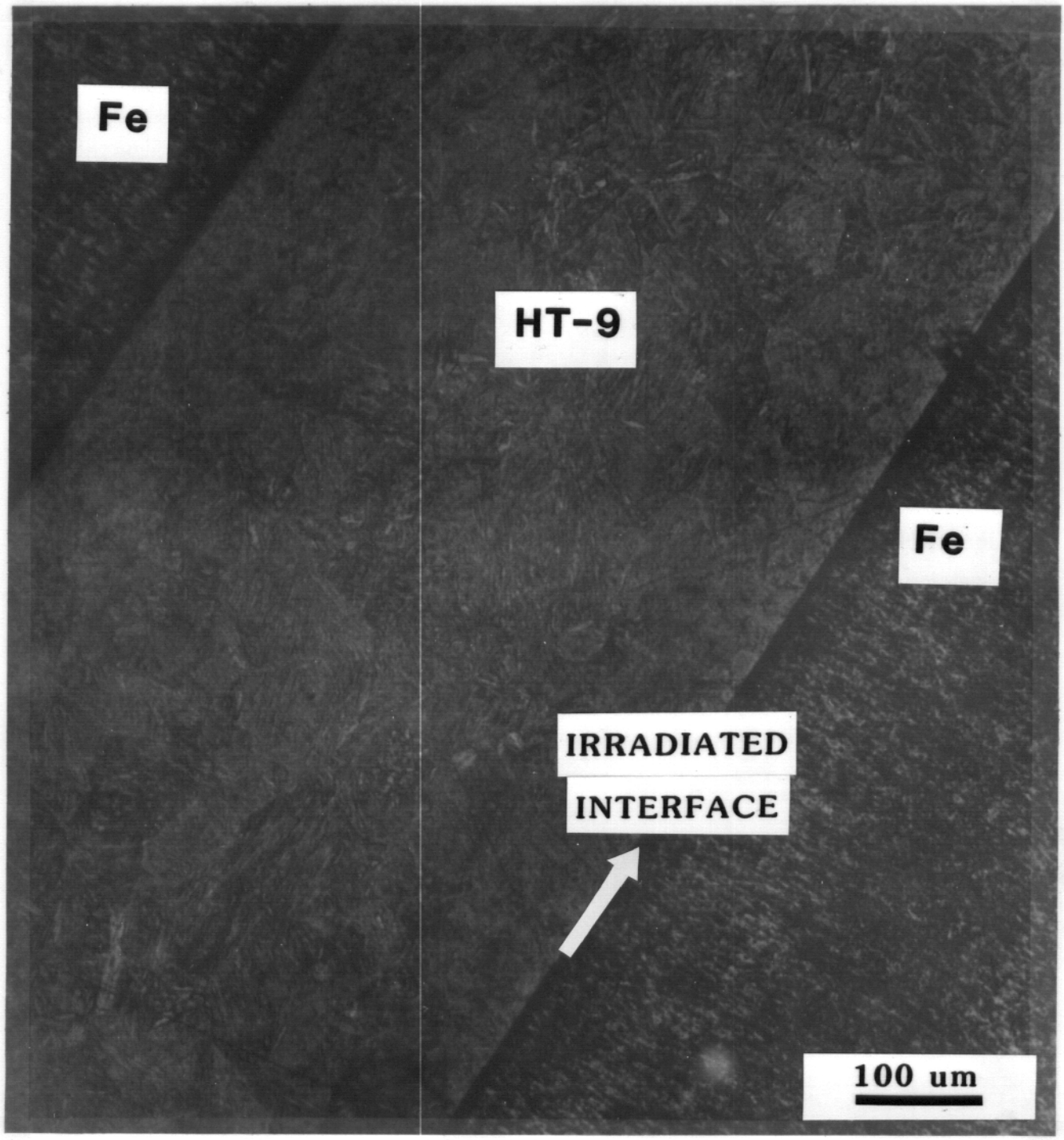

Fig. 6. Optical micrograph of an iron plated HT-9 cross-section TEM specimen.

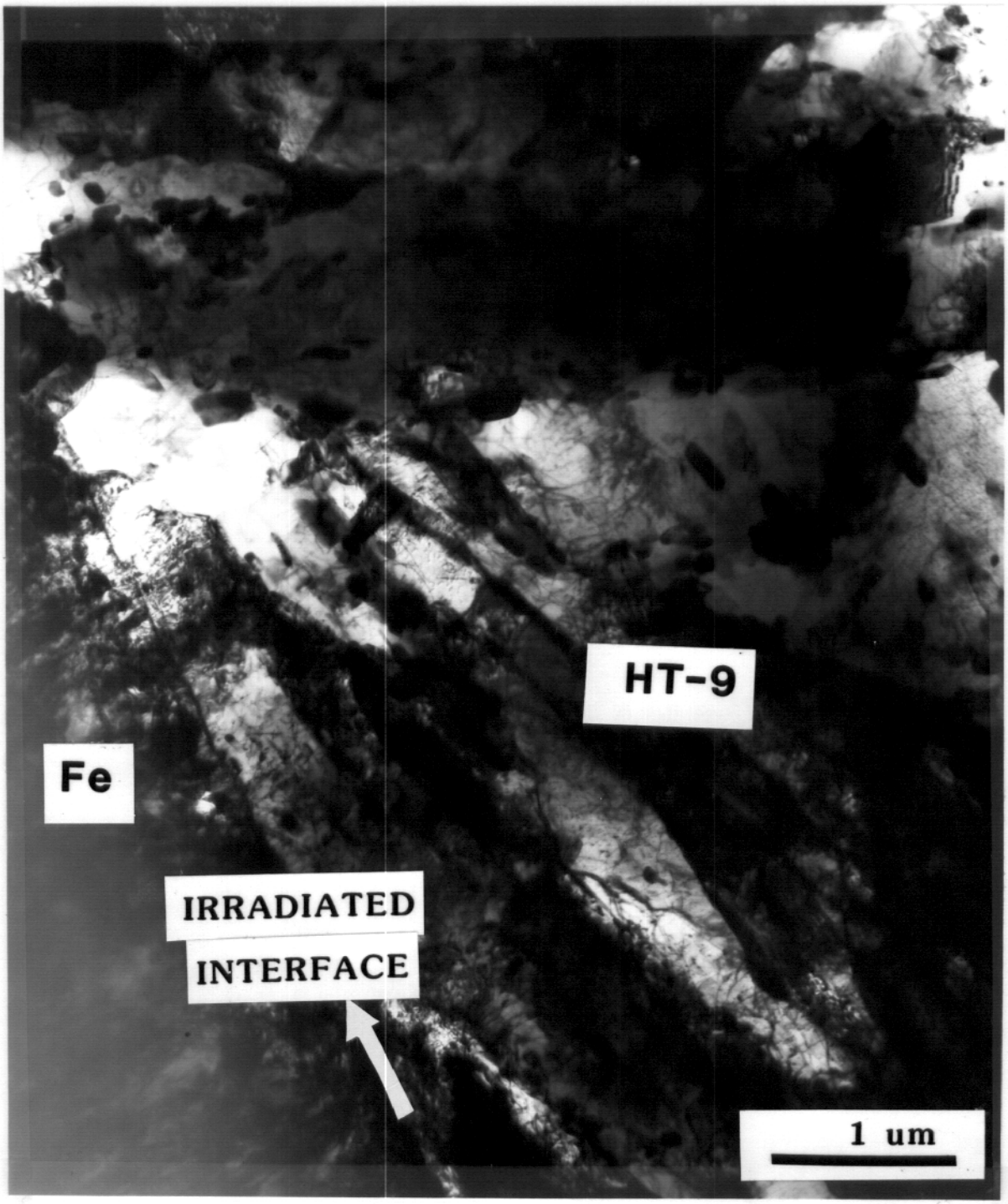

Fig. 7. A low magnification TEM micrograph showing the extent of this area along the irradiated interface of a cross-sectioned HT-9 specimen.

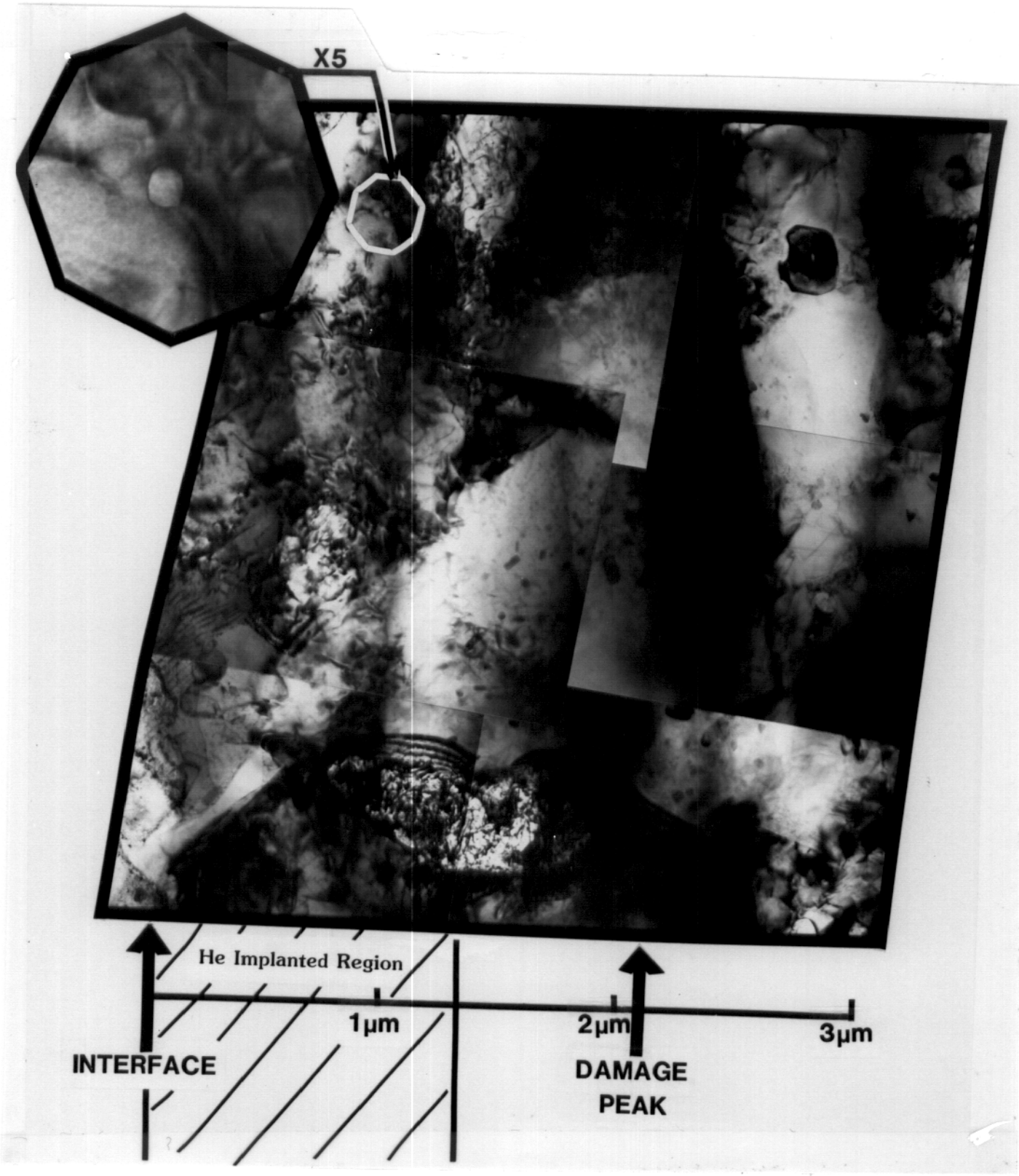

Fig. 8. A high magnification TEM micrograph showing the microstructures of a cross-section HT-9 specimen.

1/4 Cr-1 Mo steels both are already somewhat magnetic, this is a situation that one must live with. It is observed that the thinner the specimen, the better the operation condition in a TEM.

Acknowledgement

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