NISTIR 4502

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Summary

We have examined the failed or "mishap" section, and other selected sections made from 17-7 precipitation hardening stainless steel, and have concluded that the unusually large amount of retained austenite, greater than 7%, measured on the surface appears to have been produced during both the solution annealing and austenite conditioning steps of the heat treating process, but was revealed only after the austenite conditioning step. We suspect that during the heat treating process these sections may have been exposed to a furnace atmosphere that contained too much carbon or nitrogen. This exposure could have led to an increase in the amount of retained austenite both on the surface and internally.

Introduction

At a meeting on or about June 27, 1990, with Navy representatives Mr. D. Barrett and Ms. E. Bensinger, a failure of an arresting gear support assembly taken from a T-2C aircraft was discussed. At that meeting we received the following pieces of a "hat" section, through which the tailhooks are attached to the support skins on the aircraft fuselage. The sections were all identified as 17-7 precipitation hardening stainless steel, and described as follows:

1. Section that was not the "mishap" piece, but Navy stated that it had failed after installation. This part was termed an installation failure and was designated Z1 by NIST.

2. Fabricated, but never installed. Navy considers this piece not acceptable. Designated as Z2 by NIST.

3. Piece of section Navy considers satisfactory. Designated Y by NIST.

On or about August 2, 1990, Ms. Bensinger sent us part of fourth section:

4. "Mishap" piece. Also identified as "mishap" by NIST

At the initial meeting, we also received the report by Bensinger and Johnson titled "Failed North American Aviation 249-31736 Arresting Gear Hat and North American Aviation 288-310655 Arresting Gear Support Skin From T2C Mishap Aircraft 158596." We were asked to review the report and comment on the probable cause of the hat failure.

After reviewing the report and, in particular, examining the mechanical property and microstructural results, we concluded that their x-ray diffraction (XRD) findings suggested a probable cause of the "mishap" failure. The Navy found 20% retained austenite on the surface of the "mishap" piece. This amount of retained austenite is considerably higher than that normally found (7%) both on the surface and within properly heat treated 17-7PH stainless steel. The presence of excessive retained austenite has been found to reduce the mechanical properties of steel (1). We, therefore, concluded that the failed section likely had been heat treated improperly. After staff discussions, we also concluded that we did not have enough evidence or failed material to perform a complete failure analysis of the "mishap" section. Hence, with the data and the samples submitted, we directed our efforts toward determining whether the failed, "mishap" piece had been heat treated properly, and if not, what step or steps in the heat treatment cycle may have produced the excessive retained austenite.

Metallurgy of 17-7 Precipitation Hardening Stainless Steel

17-7PH steel is a stainless steel, nominally 0.09 wt. % C, 17.0 wt. % Cr, 7 wt. % Ni, and 1.0 wt. % Al, that possesses both a remarkably high ultimate strength (180-215 ksi), a high yield strength (165-200 ksi), and corrosion resistance. A minimum elongation in 2 inches for this steel in the TH1050 condition is about 6%, but values of 12% have been reported. Normally stainless steels (nominally 18 wt. % Cr and 8 wt. % Ni) and low carbon steels (less than .20 wt. % C), do not possess exceptional strength properties, but 17-7PH steel obtains these properties from a combination of the solution annealing, austenite conditioning, and precipitation hardening treatments discussed below.

The first heat treatment in the strengthening process is the solution annealing treatment. For material of the thickness of the hat section, the specification requires that the material be held at 1950 F ± 25 F under vacuum for eight minutes, and then air cooled. This exposure allows carbon and other elements to be taken into solid solution. They are held in solution by rapid cooling. This results in a stable austenitic structure; that is, the M_s, or the temperature at which martensite forms, is below room temperature. The microstructure of a properly solution annealed material consists of about 80 to 90% austenite and 10 to 20% delta ferrite stringers dispersed uniformly throughout the austenite. Ferrite that exists with austenite at high temperatures is called delta ferrite. Delta ferrite is always present regardless of the heat treatment because of the chemical composition of the steel. The amount of delta ferrite present in the austenite matrix is dependent on the solution annealing temperature. As this temperature increases, the percentage of austenite decreases while the percentage of the delta ferrite increases. The appropriate amount of delta ferrite and the prior austenitic grain size, both developed in this heat treating step, are two factors that influence the amount of retained austenite present in the steel.

The second heat treatment in the strengthening process is the austenite conditioning treatment. The solution annealed material is heated to 1400 F in a vacuum, held at that temperature for 90 to 120 minutes, cooled in air to below 60 F within one hour, and finally held at or below 60 F for at least ½ hour. During this treatment, the austenite is subjected to a temperature (1400 F) that allows the carbon and chromium to precipitate out of solid solution as chromium carbides ($Cr_{23}C_6$) - first at the delta ferrite-austenite interfaces, and then at the austenite grain boundaries. The removal of the carbon and chromium from the austenite matrix makes the austenite unstable after cooling, i.e., the loss of carbon and chromium raises the M_s which leads to the formation of untempered martensite upon cooling.

The complete transformation of the austenite to martensite is dependent upon the amount of delta ferrite present and the grain size of the steel. As stated, the austenite will be made unstable after cooling by the precipitation of carbon and chromium as chromium carbides at the delta ferrite interfaces and at the austenite grain boundaries. If there is an insufficient amount of delta ferrite present, the chromium carbides will have limited sites upon which to precipitate. Also if the grain size is too large, the carbon and chromium at the center of the austenite grains can not precipitate as grain boundary carbides in the amounts needed to make the austenite unstable. Both of these mechanisms lead to an abnormal amount of carbon and chromium in the austenite grains, making the austenite stable, and subsequently lowering the M_s temperature and producing higher amounts of retained austenite.

Another possible source of excessive retained austenite in this steel is the medium or atmosphere used. If, during either the solution anneal or the austenite conditioning treatments, the steel is exposed to an atmosphere rich in either carbon or nitrogen, the presence of these elements on the surface increases dramatically. This increase makes the austenite stable, which is desirable in the solution annealing treatment but undesirable in the austenite conditioning treatment, and consequently lowers the M_s. The lowering of the M_s in the austenite conditioning treatment results in an increase in the amount of retained austenite on the surface after cooling.

The third and final step in the heat treating process is the precipitation hardening treatment. The solution annealed and austenite conditioned material is heated to 1050 F ± 10 F for 90 to 120 minutes. At this temperature, two primary strengthening mechanisms occur. Untempered martensite is tempered, which leads to increased toughness, ductility, and corrosion resistance. Also it is reported that the aluminum in these steels is in a supersaturated solid solution after the first two steps, but at the precipitation hardening temperature, the aluminum in the martensite is precipitated out as an Al-Ni intermetallic compound. This action, together with the tempering of the martensite, leads to enhanced toughness, high strength, and high hardness.

In properly heat treated 17-7PH stainless steel, supplied in the TH1050 condition, the microstructure on the macro scale consists of tempered martensite, delta ferrite, and about 7% retained austenite. On the fine scale, precipitates of Al-Ni intermetallics should also be seen.

Experimental Procedure

The object of the investigation was to determine whether the "mishap" section had been heat treated improperly. In order to observe microstructures representative of properly heat treated material, samples were sectioned from an as-received piece and heat treated according to military specification MIL-H-6875 (Heat Treatment of Steels, Aircraft Practice, Process for). For reference purposes, photomicrographs were taken after each heat treatment. Representative photomicrographs were also taken of the as-received specimens both at the surface and at mid-thickness. The percentage of retained austenite was determined using x-ray diffraction methods on both the as-received surface, and after about 0.006 inch was removed from the surface. For check purposes, Mössbauer techniques were used on selected samples to determine the percentage of retained austenite. Metallographic samples were examined using back scattered electron imaging and x-ray mapping methods for chromium carbides both within the grains and along grain boundaries. Sections were also removed from selected specimens and the chemical composition determined.

Specified Heat Treatment

The sections, including the "mishap" piece, were to be heat treated according to military specification MIL-H-6875.

For information purposes, the specified heat treatment is summarized here. The specification states that material 0.050 inch in thickness shall be solution annealed at 1950 F ± 25 F for no more than 8 ± 0.8 minutes.

The atmosphere used for heat treating shall be either air, vacuum, argon, helium, or dry hydrogen. It had been reported that a vacuum furnace was used to heat treat the "mishap" piece, hence our heat treatments were conducted in a vacuum furnace.

The "mishap" or failed piece was to be heat treated to Lockheed specification, C-0507G (similar to MIL-H-6875), entitled "Heat Treatment of Precipitation Hardening Corrosion Resistant Steels." Table V of the Lockheed specification states that the heat treatment for 17-7PH steel, when supplied in condition TH1050, shall be as follows:

1. Solution Treating,
   (Annealing) (3)

As received (Condition A, i.e., when annealed material is required) or, as for the "mishap" section:

1950 F ± 25 F for 3 minutes plus 1 minute for each 0.010 inch of thickness (4). Cool per (5).

2. Austenite Conditioning (3).

1400 F ± 25 F for 90-120 minutes, followed by:

3. Transformation Cooling

Cool in air or water to below 60 F within 1 hour of austenite conditioning; hold for ½ hour minimum.

4. Precipitation Hardening (3)

1050 F ± 10 F for 90-120 minutes

Reference (3) states that the recommended heat up times are given in Table IX. Table IX does not give a recommended heat up time for material that is 0.050 inch thick. The table does give a maximum heat up time of 20 minutes for steel ¼ inch thick. The reference also states that in all cases soaking time shall not begin until parts or material reach specified temperature. Reference (4) states that time at temperature shall be held within +10%, -0%, and reference (5) states that the material shall be air cooled in a place having free air circulation or oil quenched.

RESULTS

Chemical Composition

The chemical compositions of selected pieces, as determined by an independent source, are shown in Table 1. The results indicate that the steel is within the composition limits for 17-7PH stainless steel.

Microstructure

Specimen Heat Treated According to Lockheed Specification C-0507G

A specimen was removed from section NIST-Z1 and vacuum heat treated according to the Lockheed specification C-0507G. This section was not the "mishap" piece, but one that failed after installation. Photomicrographs were taken of the microstructure after each heat treating step. For future reference this heat treated sample will be identified as the "standard" specimen.

The microstructure observed after solution annealing is shown in Figure 1. The microstructure consisted primarily of austenite (light-gray background) and delta ferrite (darker particles). Note the absence of chromium carbide precipitates around the delta ferrite particles. Due to etching conditions, the austenite grain boundaries are not visible in these photomicrographs.

The specimen was given the austenite conditioning treatment and the resultant microstructure shown in Figure 2 consisted of delta ferrite, and austenite grains. The dark particles are the delta ferrite, and the austenite grains the light and gray areas. Surrounding the delta ferrite particles are the chromium carbide precipitates. The variance in gray tone of the austenite grains reflects to different austenite grain orientations.

Figure 3 shows the microstructure for the steel after the precipitation hardening treatment. The microstructure consisted of tempered martensite (dark needle-like regions), some delta ferrite (light-gray spherical areas), and unresolvable retained austenite. The prior austenitic grain size was ASTM number 11.5, which corresponds to an average grain diameter of 6.7μm.

Photomicrographs of the As-Received Specimens

The microstructures observed near the center of the as-received specimens NIST-Y, NIST-Z1, NIST-Z2 are shown in Figures 4, 5, and 6. Figures 4a and 4b show the microstructures observed for sample NIST-Y. (This sample was reported to be acceptable by the Navy.) The microstructure consisted of stringers of delta ferrite in a tempered martensite matrix. Note in Figure 4b the chromium carbide precipitates surrounding the delta ferrite particles. The prior austenite grain size appeared to be quite uniform and the grain size number was 12.1, which corresponds to an average grain diameter of about 5.6μm.

Figures 5a and 5b show the microstructures observed for specimen NIST-Z1. (Sample NIST-Z1 was reported to be the piece that had failed after installation.) The photomicrographs of this sample revealed that the steel did not contain the number of delta ferrite stringers or the amount of tempered martensite observed in either the "standard" or NIST-Y samples. The delta ferrite particles appeared to be surrounded by chromium carbide precipitates. The prior austenitic grain size was number 11.4, which corresponds to an average grain diameter of 7.0μm.

Figures 6a and 6b show the microstructures observed near the center of sample NIST-Z2. (The section from which this sample was taken had not been installed, but the Navy considers unacceptable.) The microstructure was similar to that observed in sample NIST-Z1, the section that had failed after installation. A small amount of delta-ferrite stringers and tempered martensite was observed. Surrounding the delta ferrite were also what appears to be chromium carbide precipitates. The prior austenitic grain size was also ASTM number 11.4, which corresponds to an average grain diameter of 7.0μm.

Photomicrographs of the "Mishap" Piece

Figures 7 and 8 show the microstructures obtained for the as-received "mishap" piece. Figure 7 shows that the microstructure contained a reduced amount of both delta ferrite and tempered martensite compared to the "standard" specimen. Figure 7b, the same area as Figure 7a but at a higher magnification, shows that the microstructure consisted of delta ferrite (white particles) and needle-like martensite (dark particles). The decrease in the number of delta ferrite particles indicated that chromium carbides had fewer sites on which to precipitate. Figures 8a and 8b show photomicrographs taken at different locations in the "mishap" piece. Delta ferrite and tempered martensite were observed, but not in the amounts observed in either the "standard" or NIST-Y samples. These photomicrographs revealed the absence of chromium carbide precipitates on the delta ferrite/austenite interfaces. The prior austenitic grain size was ASTM number 10.0, which corresponds to an average grain diameter of 11.0μm.

Grain Size

Grain size measurements were conducted on the specimens using the ASTM intercept method. The results are shown in Table 2. The results indicate that the grain size of the "mishap" specimen was different than the other specimens. The prior austenitic grain size for the "mishap" specimen was ASTM number 10.0 which corresponded to an average grain diameter of 11.0μm. The average prior austenite grain size diameter for the other specimens was 6.5μm. This corresponds to an ASTM number of about 11.6.

Back Scattered Electron Imaging and X-Ray Mapping Results

The previous discussion of the factors that determine the stability or instability of austenite concluded that after austenite conditioning treatment, if there were a limited number of chromium carbide precipitates at the interfaces of the delta ferrite/austenite boundaries and within the grain boundaries, the austenite would be stable, the M_s lowered, and the microstructure would contain an appreciable amount of retained austenite. In order to examine this hypothesis, back scattered electron and x-ray techniques were used to probe these areas of the "mishap" and "standard" specimens. Figure 9 shows photomicrographs of the "standard" specimen. Chromium carbides were observed along the boundaries of the delta ferrite particles. Figure 10 shows photographs of the "mishap" specimen. In figure 10a there was evidence of grain boundary precipitation of the chromium carbides, but in figure 10b evidence of chromium carbide precipitation around the delta ferrite particles was inconclusive, hence our hypothesis could not be proven.

X-Ray Diffraction and Mössbauer Results

Table 3 shows the x-ray and Mössbauer results. The determinations were conducted on as-received, as-polished, and chemically polished surfaces by the Navy, NIST, and an independent laboratory. Consistent results were obtained for the surface of the "mishap" specimen; that is, all of the measurements showed that the "mishap" specimen's surface contained an abnormal amount of retained austenite, greater than 20%. Similar results, with the exception of NIST's, were found for NIST-Z1's surface. All of the investigators found the as-received surface of NIST-Y, the specimen the Navy contends was acceptable, contained an acceptable amount of retained austenite, 7% or less. When approximately 0.006 inch was removed by chemical polishing from the surface of the "mishap", NIST-Z1 and Z2 specimens, a decrease in the amount of retained austenite was observed. The reasons for the scatter in retained austenite measurements were not investigated. For example NIST's determination of sample Z1 and the independent source's determination of Z2 seem to vary significantly from the other three measurements. In our opinion the agreement of at least three measurements, combined with the metallographic results, is sufficient evidence to support the claim of this report. Based on the results shown in the table, it appears that retained austenite measurements should be taken at a number of areas on the sample in order to obtain a value characteristic of the piece.

Additional Metallographic Examinations

Because of the variation found by x-ray of the surface versus the interior, additional metallographic examinations were performed. One of the characteristics of retained austenite is that it is not attacked by etchants which attack the other constituents of the microstructure. The "mishap", NIST Z1, Z2, and Y specimens were etched in Villela's solution for an extended period, and then microscopically examined at high magnification. Figures 11 and 12 show the microstructures observed near the surface and along the center line of the "mishap" specimen. The white areas in the photomicrographs are retained austenite. Similar results, Figures 13 and 14, were observed for specimens NIST Z1 and Z2.

The microstructures observed on the surface and along the center of specimen NIST Y were not the same as those observed on the surfaces of the "mishap," NIST Z1 or Z2 specimens. Figure 15 shows the microstructure observed near the surface and center of NIST-Y. Examples of the limited amounts of retained austenite are shown by the arrows in the photomicrograph.

Similar microscopic examinations were conducted on the "standard" specimen, that is the specimen that was heat treated according to the Lockheed specification. Figure 16 shows the microstructures observed near the surface and center of this specimen. An example of the retained austenite found in the microstructure is shown by the arrows in the photomicrograph.

Discussion of Results

The "mishap", NIST-Z1, NIST-Z2, and NIST-Y samples of 17-7PH stainless steel were metallurgically examined. The bulk chemical compositions of selected specimens were determined and the results indicated that the steel was within the limits for 17-7PH stainless steel.

For comparison purposes, a sample was sectioned from Z1 and heat treated in a vacuum furnace according to the specification. This sample was identified as the "standard" specimen. Photomicrographs taken after the solution anneal, the solution anneal plus austenite conditioning, and after solution annealing plus austenite conditioning plus precipitation hardening provided representative microstructures of each treatment. Photomicrographs of the solution annealing treatment revealed that the microstructure consisted of austenite with regions of delta ferrite. The microstructure after the austenite conditioning contained particles of delta ferrite surrounded by chromium carbides plus austenite, and the microstructure after the precipitation hardening treatment contained tempered martensite, delta ferrite, plus some retained austenite. The microstructures were all similar to those observed in the literature for properly heat treated 17-7PH stainless steel.

The as-received specimens were examined metallographically. The "mishap" specimen was noticeably different from the others and, in particular, from the specimen that was heat treated according to the specification. The "mishap" specimen appeared to contain a large percentage of retained austenite, and small amounts of delta ferrite and tempered martensite. Retained austenite, when present in amounts greater than 7%, is reported in the literature to lead to poor mechanical properties. Specimen NIST-Z1 (not the "mishap" sample but one that failed after installation) and NIST-Z2 were also examined. The microstructures appeared similar to the "standard" specimen. Sample NIST-Y (considered to be acceptable by the Navy) was also examined, and its microstructure appeared to be similar to that of the "standard" specimen.

It was reported by the Navy that the "mishap" section had been heat treated improperly. Instead of the required 8-minute solution anneal, the section was held at temperature for 20 minutes. Exposure for this prolonged time often leads to increased grain size which could lead to an increase in the amount of retained austenite after the austenite conditioning treatment. Because of the increase in grain size and the limited amounts of delta ferrite, there was a possibility that the amount of carbon needed to make the steel unstable (i.e., to lower the M_s in the austenite conditioning treatment) could not migrate to the grain boundaries or have enough delta ferrite sites upon which to precipitate as chromium carbides. As a result there is an excessive amount of carbon left in solid solution and, upon quenching after the austenite conditioning, retained austenite, in excess of that normally observed, forms.

Considering this, the grain size of all the specimens was determined. The ASTM grain size number and average grain diameter of the "mishap" section was found to be 10 and 11.0μm, respectively. The average grain size number and grain diameter for the other specimens examined were 11.6 and 6.5μm, respectively. It was reported that during the solution anneal the "mishap" specimen was held at temperature for 20 minutes, rather than the recommended 8 minutes. It is quite possible that the extended period at the solution annealing temperature was responsible for the measured increase in prior austenitic grain size.

Selected specimens were microscopically examined using back scattered electron and x-ray imaging techniques for the presence of chromium carbides along the delta ferrite/austenite interfaces and within the grain boundaries. The expected chromium carbides were observed surrounding the delta ferrite in the "standard" specimen. On the "mishap" specimen, the carbides were only observed in the austenite grain boundaries. Thus, we concluded that the extended solution annealing of 20 minutes did increase the grain size, but not to where it resulted in an increase in retained austenite after the austenite conditioning treatment.

The as-received specimens were examined, using x-ray diffraction and Mössbauer methods, both on the surface and after 0.006 inch had been removed from their surfaces. The results showed that there was a higher percentage of retained austenite on the surfaces than on the insides of the specimens. Additional microscopic examinations were undertaken on the as-received specimens to view the retained austenite which had been measured using x-ray and Mössbauer methods. New as-received specimens were polished, etched, and viewed in cross section. The results, after using Villela's etch for an extended period, showed that the surfaces of the "mishap", NIST-Z1, and NIST-Z2 specimens did, in fact, contain excessive amounts of retained austenite.

The presence of the retained austenite on the surface was established from both the x-ray and metallographic results. The excessive retained austenite on the surface could possibly have been produced by the atmosphere to which the section was exposed during the solution annealing or austenite conditioning, but was revealed only after the austenite conditioning treatment. Normally, the sections are heat treated in a vacuum, and they have been certified as being done so. However, we suspect that even though these specimens were vacuum heat treated, they may have been subjected to an atmosphere rich in either carbon or nitrogen. Metallurgically, these elements have the ability to make austenite stable; that is, they lower the M_s temperature. With a lowered M_s temperature, martensite does not form. Of course this is ideal after the solution anneal treatment, but undesirable after the austenite conditioning treatment because after this treatment, retained austenite will form if the atmosphere is rich in carbon or nitrogen. It is this retained austenite that transforms with stress to untempered or brittle martensite with poor mechanical properties. Therefore, we suggest that quite possibly the atmosphere to which the "mishap" specimen had been exposed led to the excessive retained austenite on both the surface and internally.

TABLE 1. Chemical Composition, in Weight Percent, of Test Specimens and 17-7 PH Stainless Steel.

Element	17-7[1] PH STEEL	"MISHAP"	NIST-Z1[3]	NIST-Z2[4]	NIST-Y[2]
Carbon	0.09 MAX	0.08	0.07	0.08	0.07
Manganese	1.00 MAX	0.46	0.57	0.46	0.52
Phosphorus	0.04 MAX	0.017	0.021	0.014	0.014
Sulfur	0.04 MAX	0.011	0.010	0.012	0.014
Silicon	1.00 MAX	0.41	0.56	0.72	0.49
Chromium	16.-18.00	17.12	17.08	17.24	17.39
Nickel	6.5-7.75	7.20	7.14	7.18	7.45
Aluminum	0.75-1.50	1.03	1.07	1.03	1.00
Molybdenum	residual	0.05	0.18	0.05	0.20

TABLE 2. Grain Size Measurements.

Specimen Identification	Grain Size Number	Corresponding Diameter of "Average" Grain Section, μm
"Standard"	11.5	6.7
NIST-Y[2]	12.1	5.6
NIST-Z1[3]	11.4	7.0
NIST-Z2[4]	11.4	7.0
"MISHAP"	10.0	11.0

1. Armco 17-7 PH and PH 15-7 Mo Stainless Steel Sheet and Strip, Armco Steel Corporation, Middletown, Ohio, 1966.
2. Acceptable by Navy
3. Failed after installation
4. Navy contends is unacceptable

TABLE 3. Percentage of Retained Austenite as Determined Using X-Ray Diffraction and Mössbauer Methods.

Specimen Identity	Surface Preparation	XRD			Mössbauer
		Navy	NIST	IND Source	NIST
"MISHAP"	As Rec'd	20.3	25.3	21.9	24.0
NIST-Z1	"	22.4	12.7	34.2	19.0
NIST-Z2	"	4.1	5.0	21.0	10.0
NIST-Y	"	5.0	4.6	ND[1]	7.0
"MISHAP"	.006" removed by chemical polishing	ND	5.3	11.3	9.0
NIST-Z1		ND	4.0	10.8	10.0
NIST-Z2		ND	10.0	16.8	12.0

1. Not Determined

References

1. Castleman, L.S., Averbach, B.L., and Cohen, M., "Effect of Retained Austenite upon Mechanical Properties," Transactions of American Society for Metals, Vol. XLIV, pp. 240-263, 1952.

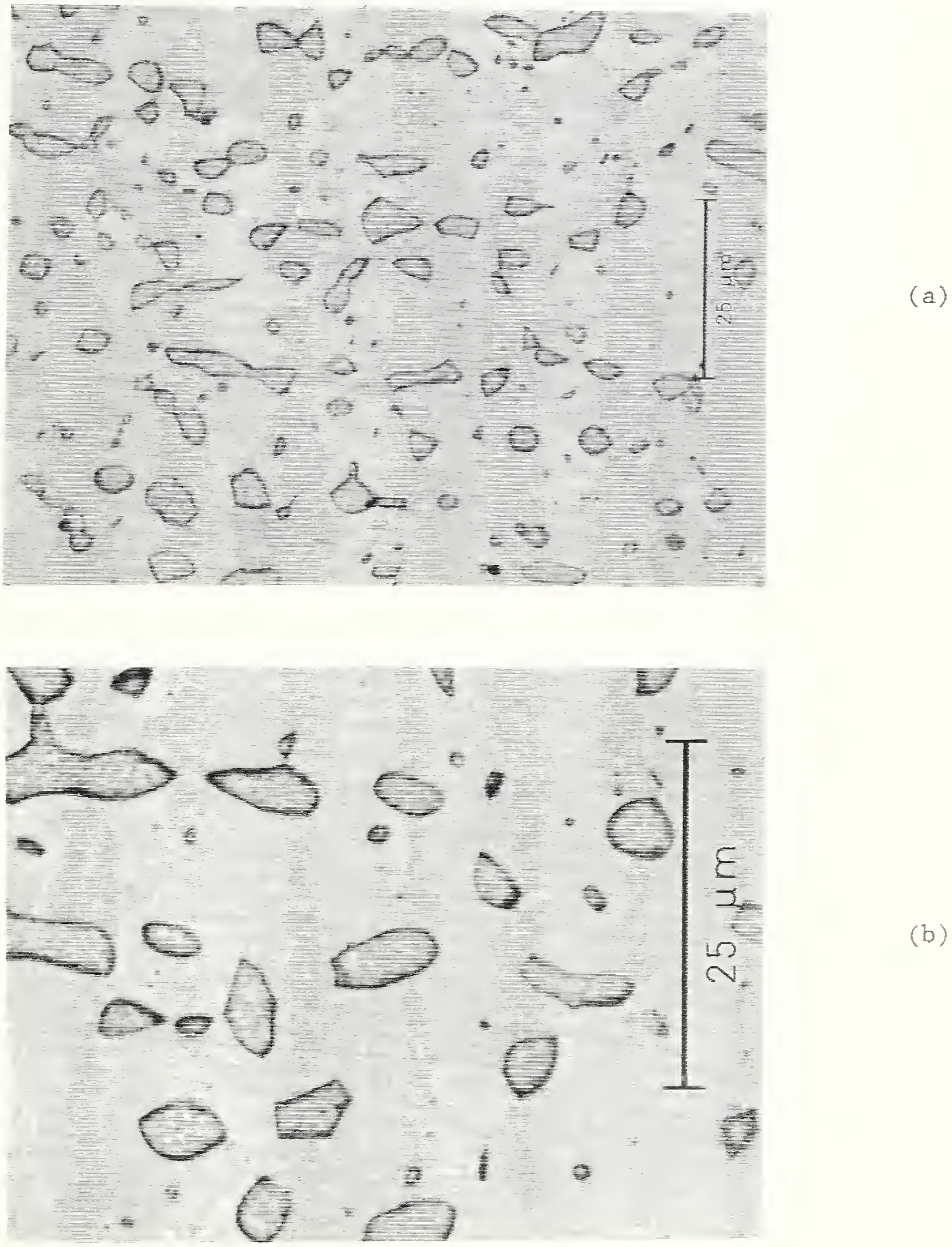


Figure 1. Photomicrographs of specimen NIST-Z1 after solution annealing in vacuum at 1950 F for 8 minutes and cooled in air. Dark particles are delta ferrite, background is austenite.
Mag: (a) X1000, (b) X2000. Etch: Villela's

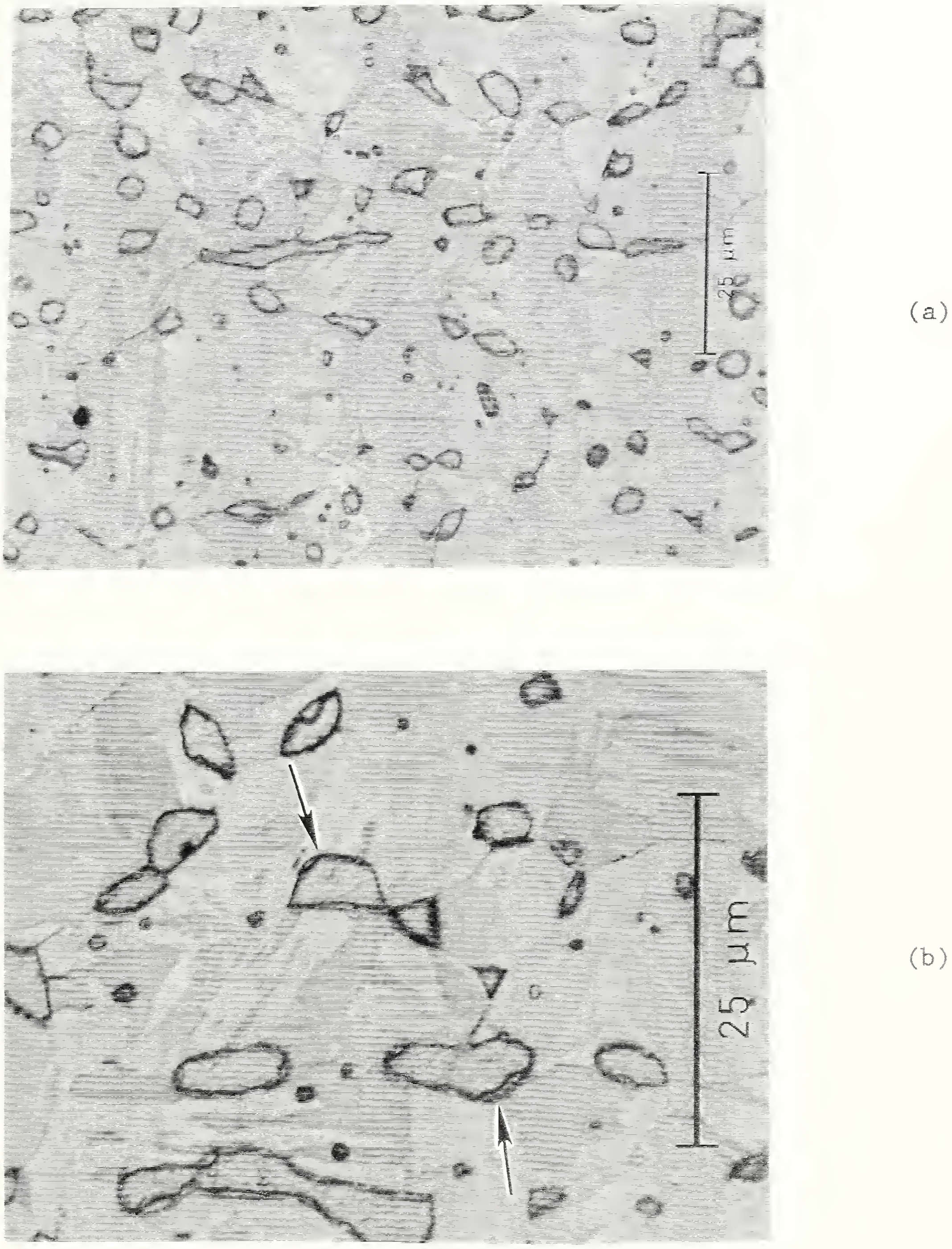


Figure 2. Photomicrographs of specimen NIST-Z1 after solution annealing, and austenite conditioning in vacuum at 1400 F for 90 minutes. The dark particles are the delta ferrite. Note the chromium carbide precipitates (arrows) surrounding the delta ferrite particles. Mag: (a) X1000, (b) X2000. Etch: Villela's

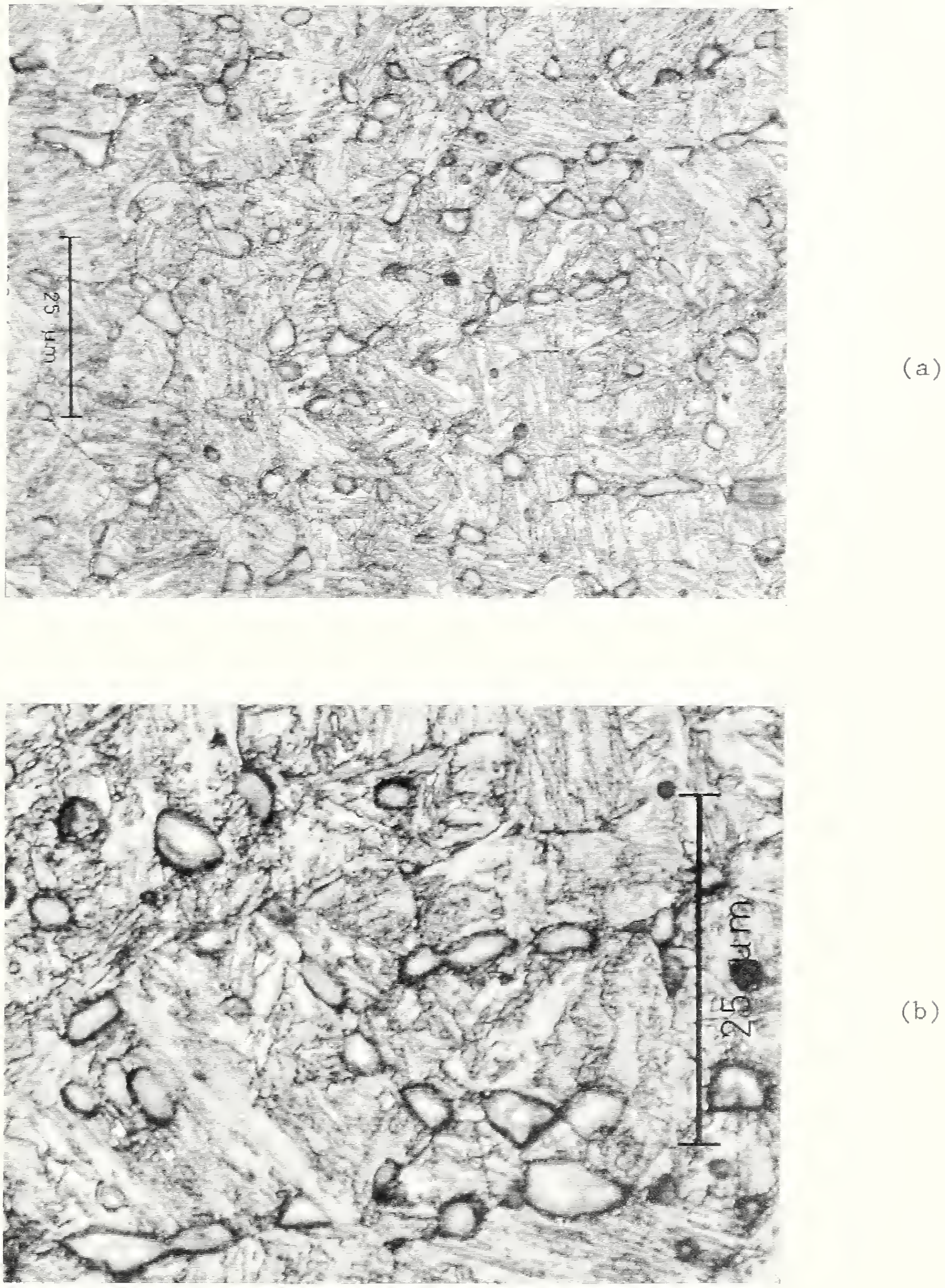


Figure 3. Photomicrographs of specimen NIST-Z1 after solution annealing, austenite conditioning, and precipitation hardening at 1050 F for 90 minutes. The needle-like dark particles are tempered martensite, and the grey particles, delta ferrite. The retained austenite is unresolvable.
Mag: (a) X1000, (b) X2000. Etch: Villela's

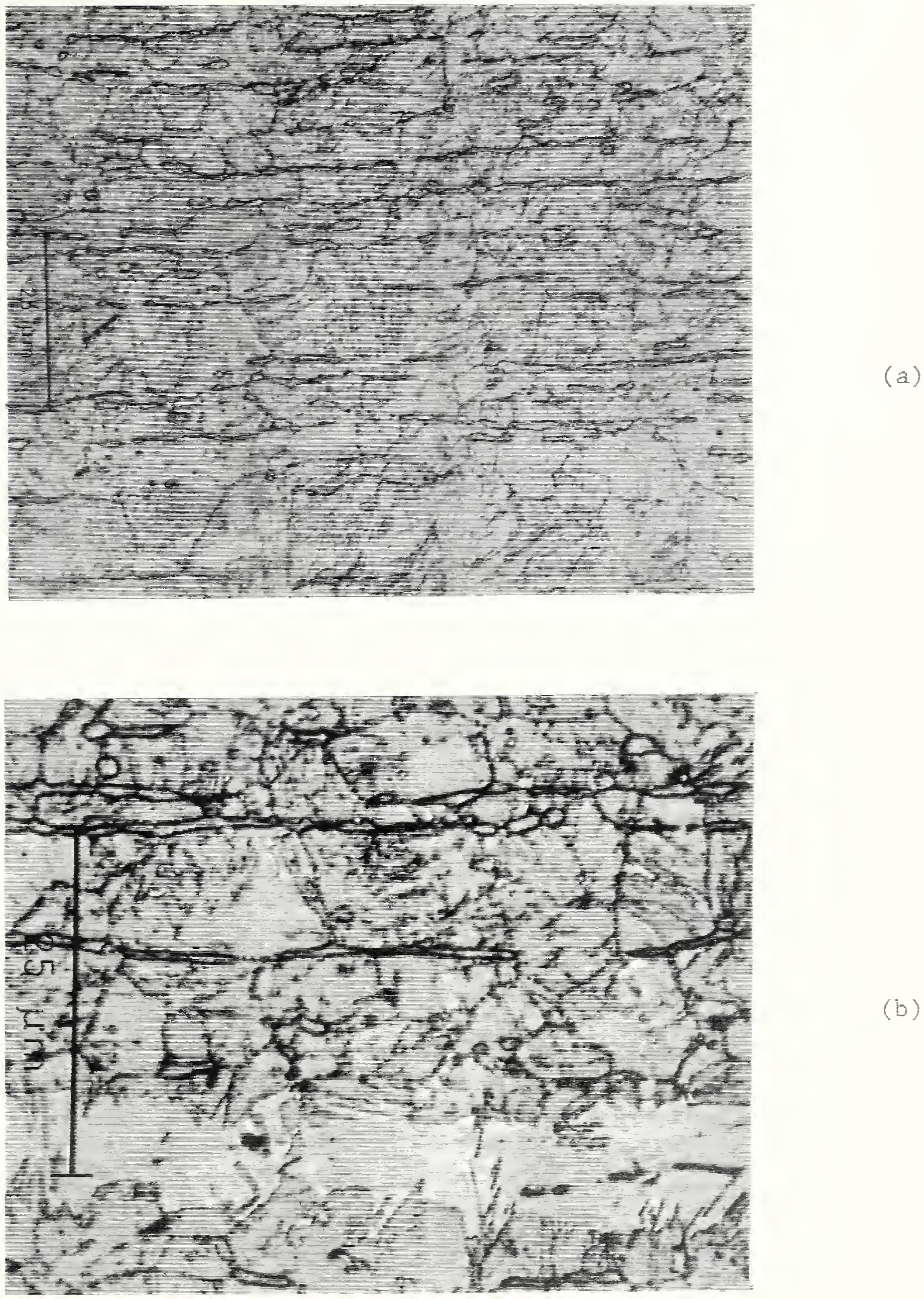


Figure 4. Photomicrographs taken near the center of specimen NIST-Y. The microstructure consists of stringers of delta ferrite and tempered martensite. The retained austenite is unresolvable.
Mag: (a) X1000, (b) X2000. Etch: Villela's

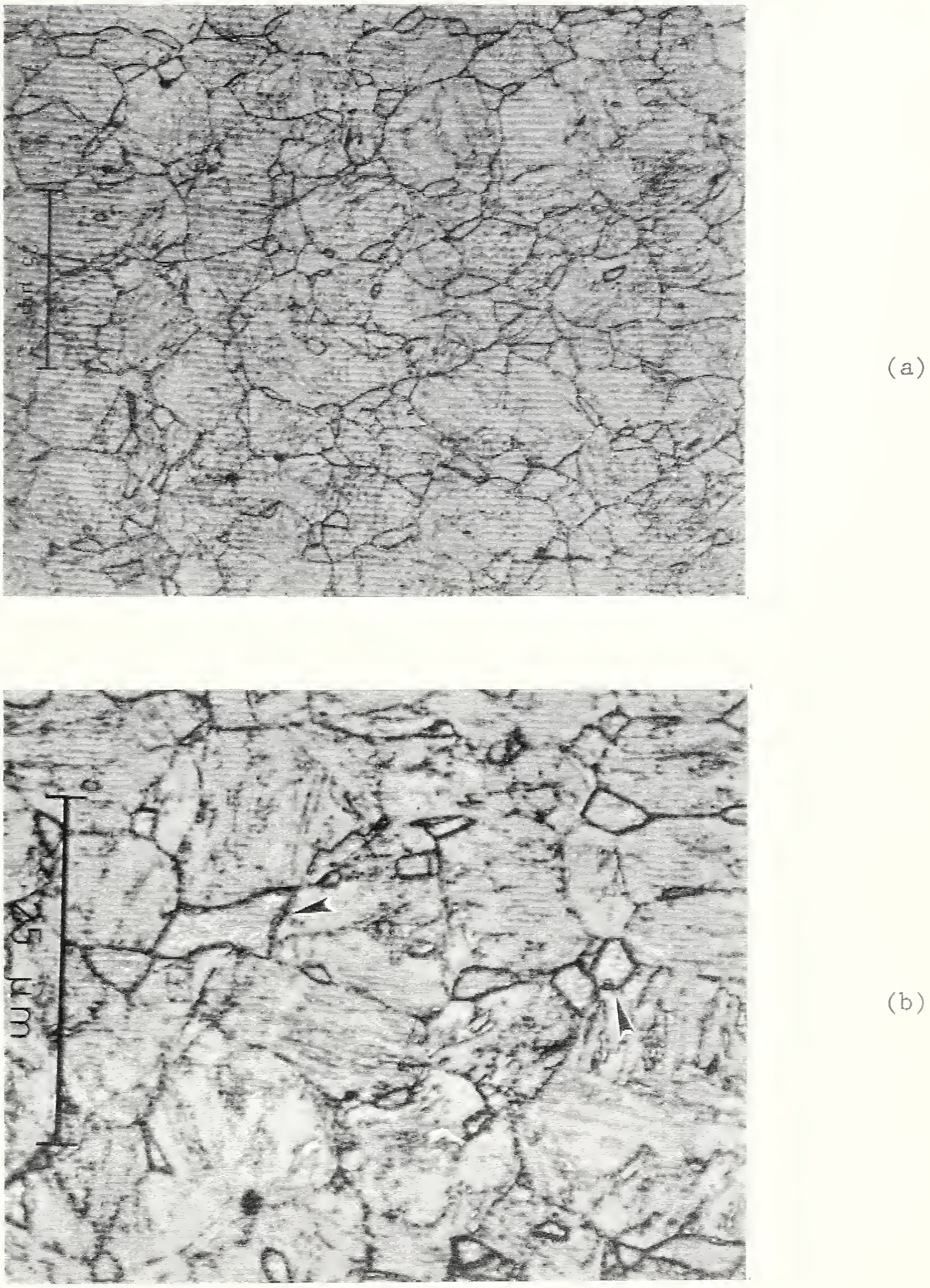


Figure 5. Photomicrographs taken near the center of specimen NIST-Z1. The section had failed after installation. Note the absence of delta ferrite in the microstructure as compared to NIST-Y. Tempered martensite (dark needles) and chromium carbides (arrows) were observed.
Mag: (a) X1000, (b) X2000. Etch: Villela's

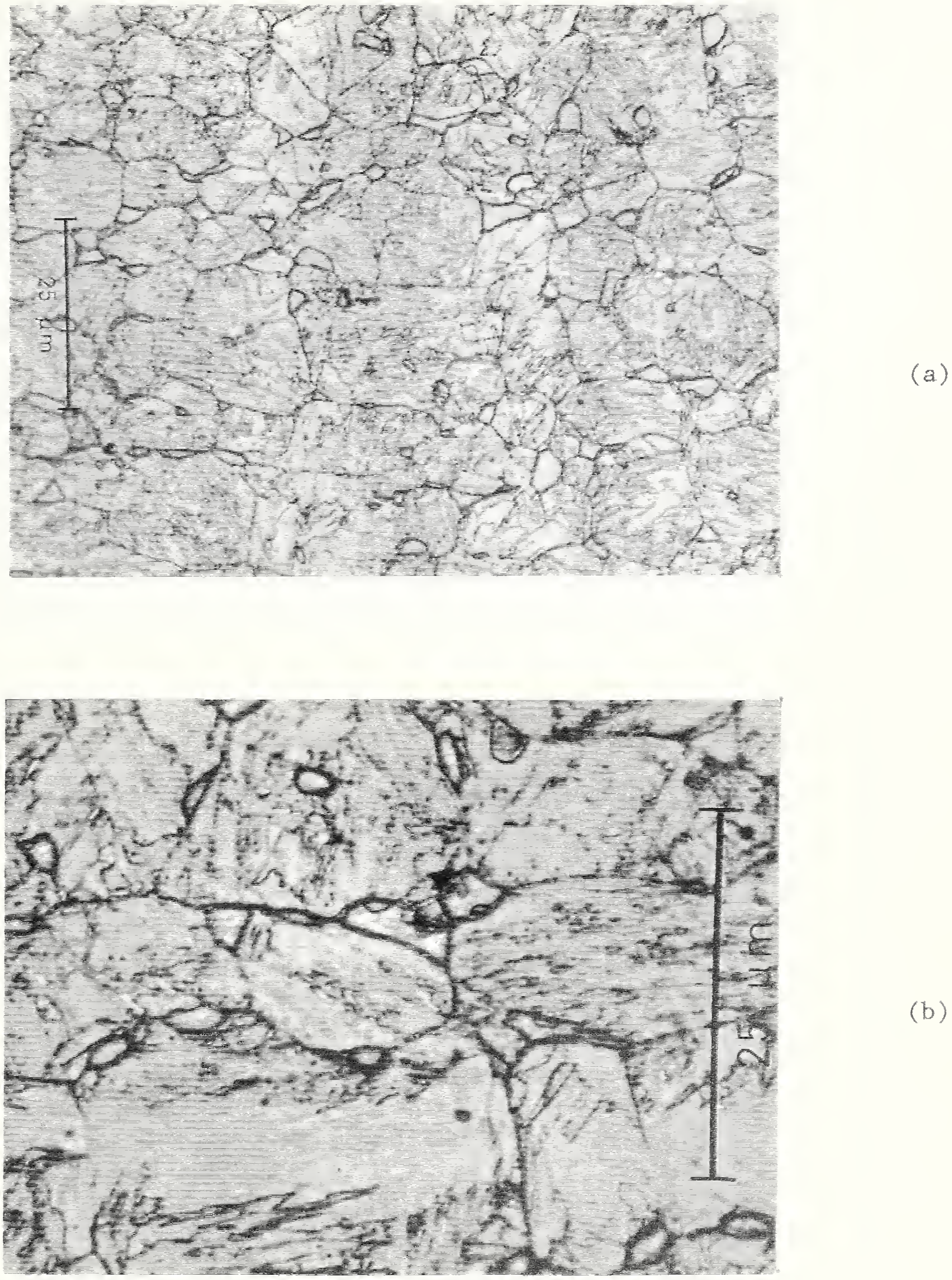


Figure 6. Photomicrographs taken near the center of specimen NIST -Z2. The Navy suspects that this sample is not acceptable for service. Note the absence of delta ferrite in the microstructure. Tempered martensite and chromium carbides were also observed. Mag: (a) X1000, (b) X2000. Etch: Villela's

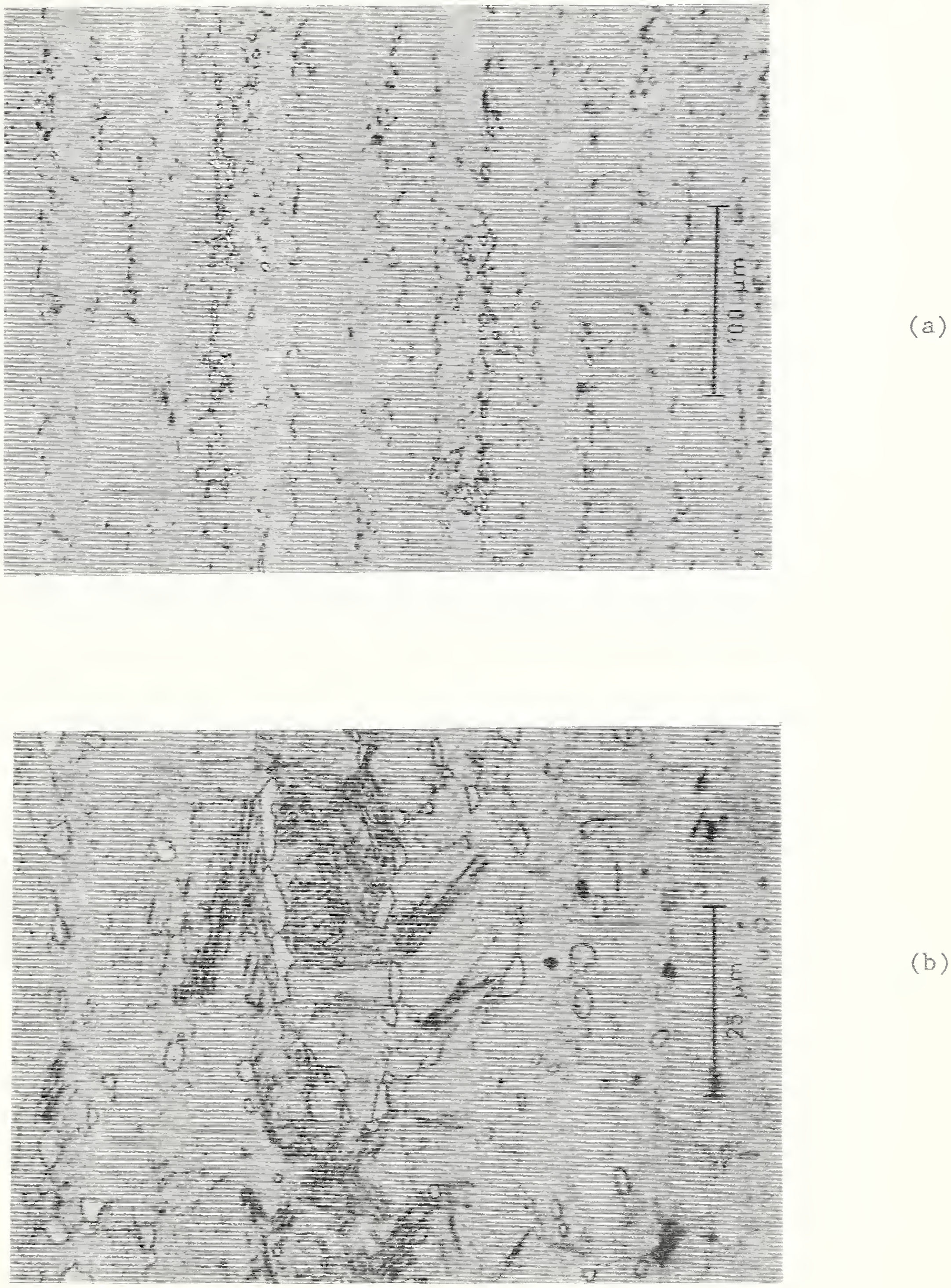


Figure 7. Photomicrographs of the "mishap" specimen. The microstructure consisted of tempered martensite (dark needles), delta ferrite (gray particles), and unresolvable retained austenite. Mag: (a) X250, (b) X1000. Etch: Villela's

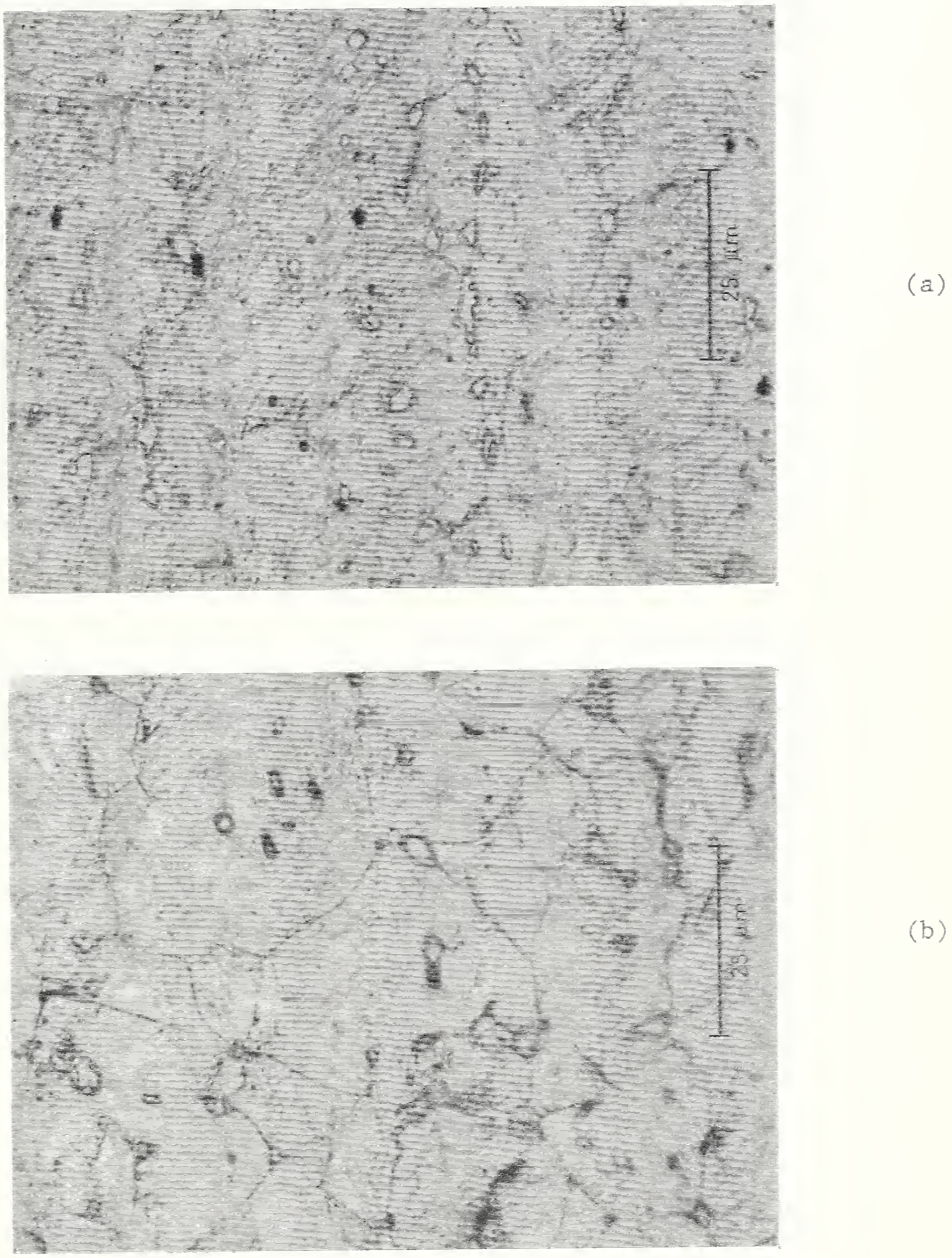


Figure 8. Photomicrographs of the "mishap" specimen taken in another area. The microstructure consisted of delta ferrite (round grey-like particles), tempered martensite, and carbides dispersed throughout the austenite matrix. Note the absence of chromium carbides around the delta ferrite.
Mag: Both X1000. Etch: Villela's

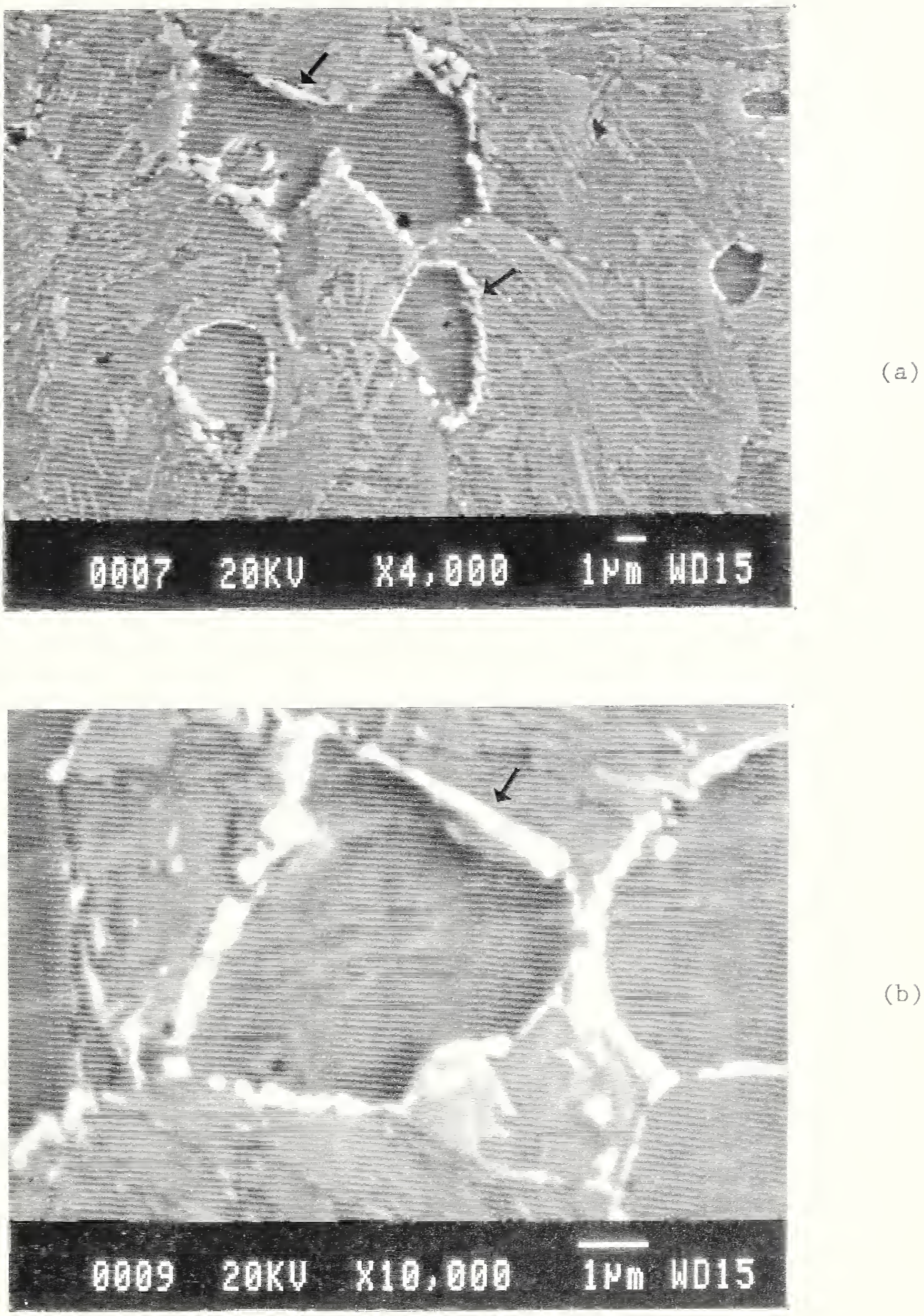
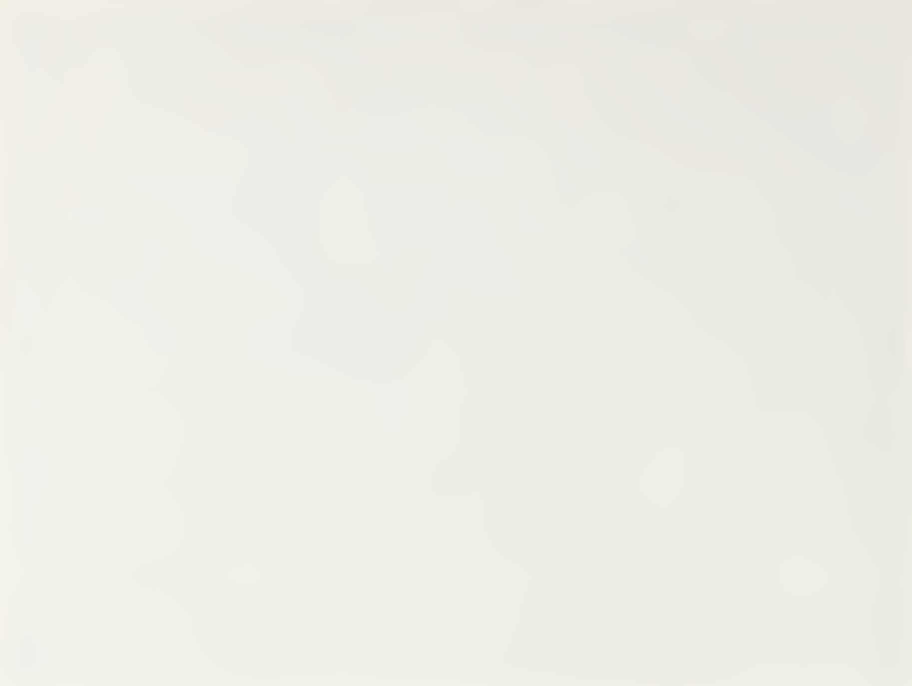


Figure 9. Photographs of the "standard" specimen showing the presence of chromium carbides (white) surrounding the delta ferrite. Mag: (a) X4000, (b) X10000.



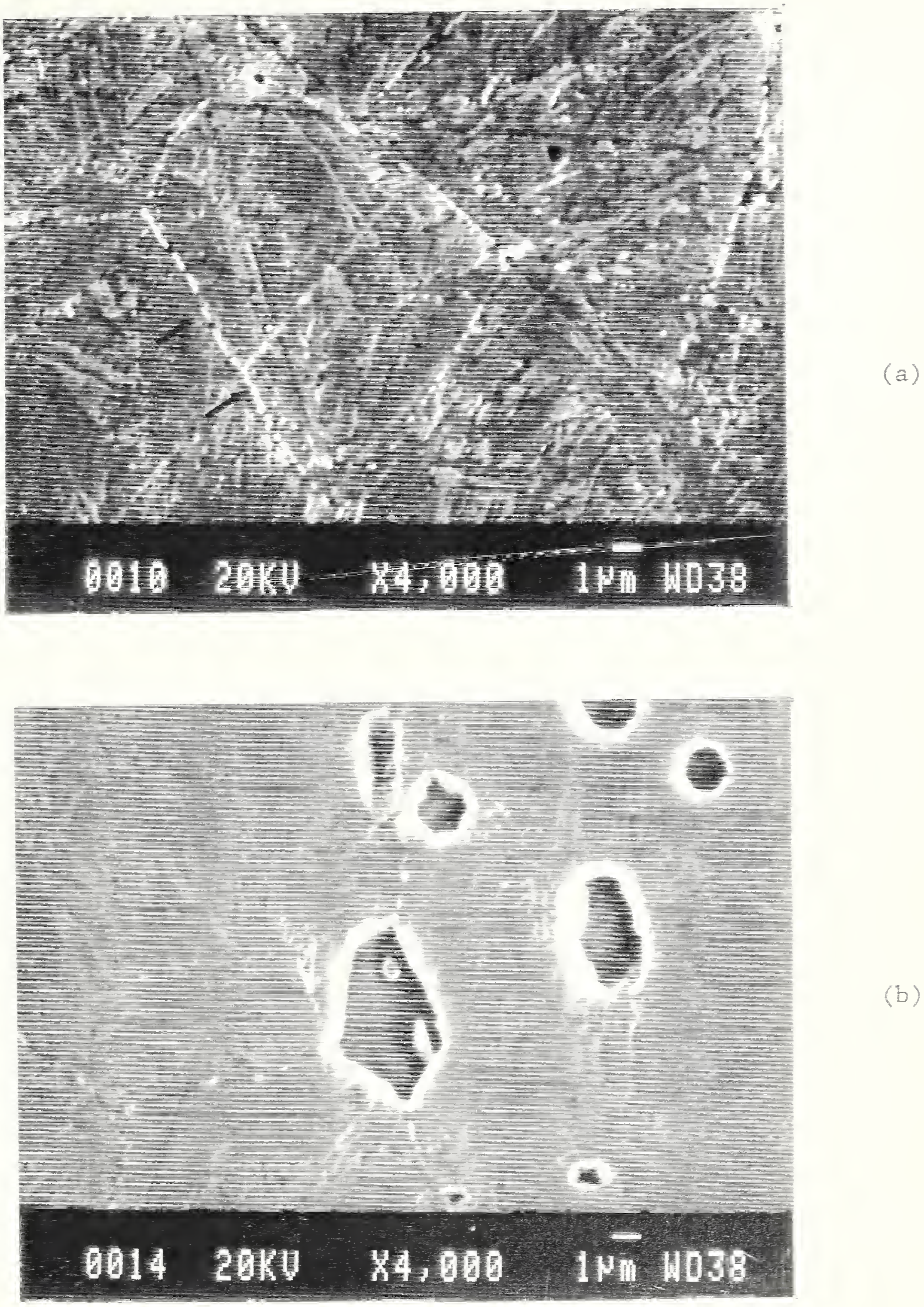


Figure 10. Photographs of the "mishap" specimen showing the chromium carbides (arrows) along the grain boundaries (a), and in (b), evidence of carbides around the delta ferrite was inconclusive.
Mag: Both X4000

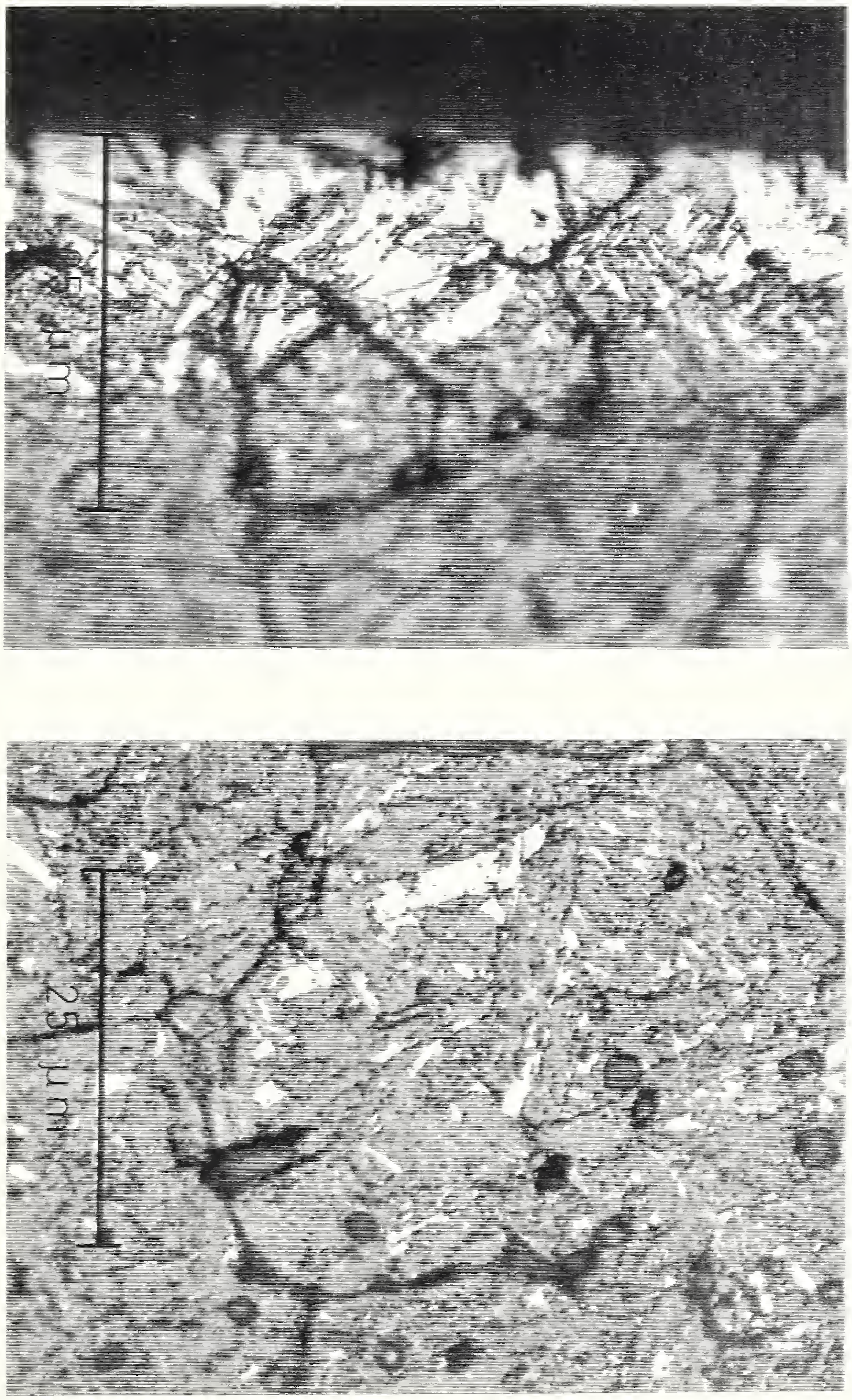


Figure 11. Photomicrographs of the "mishap" specimen taken at the surface. The white areas are retained austenite.
Mag: Both X2000. Etch: Villela's

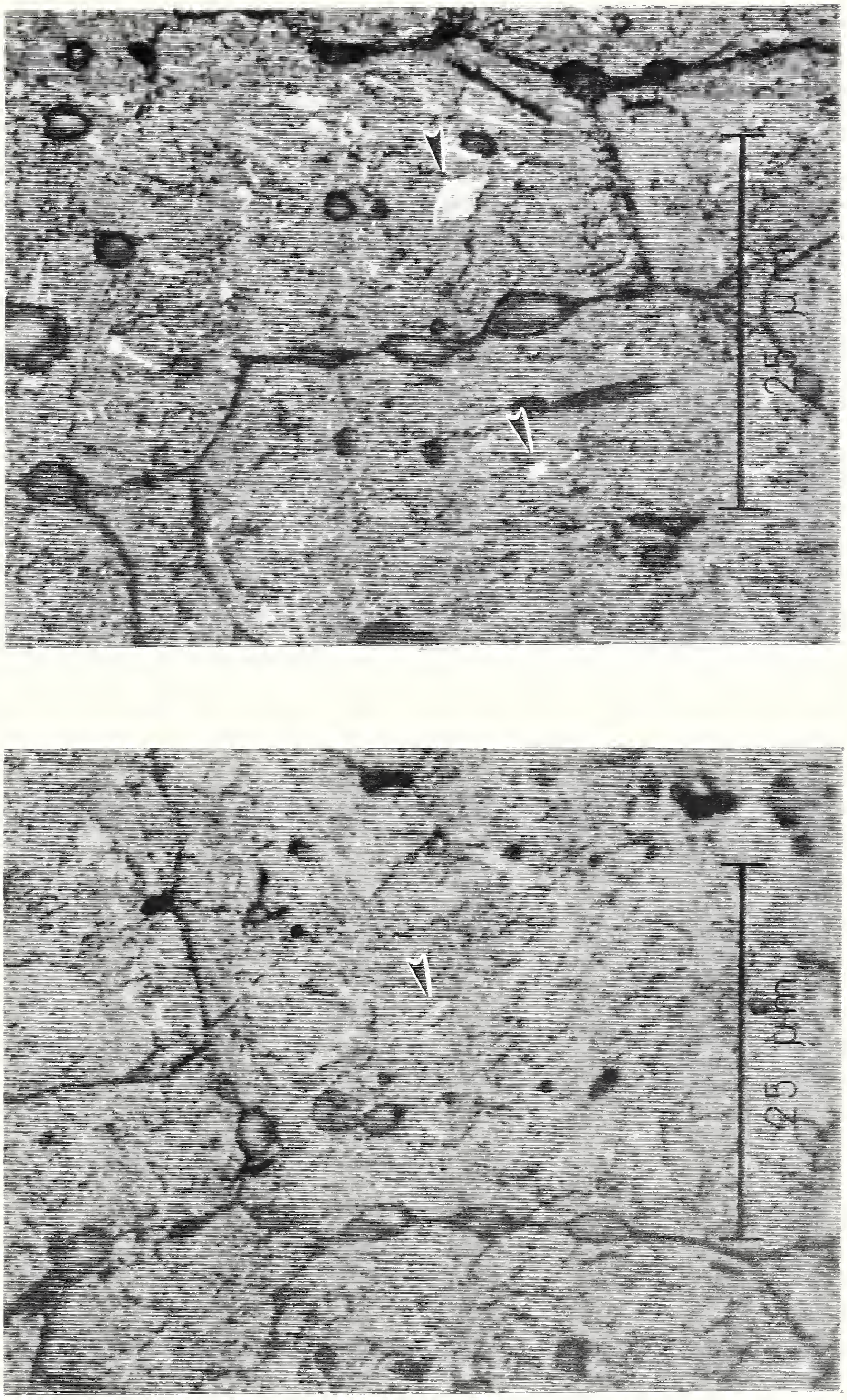


Figure 12. Photomicrographs of the "mishap" specimen taken at the center of the specimen. The white areas (arrows) are the retained austenite.
Mag: Both X2000. Etch: Villela's

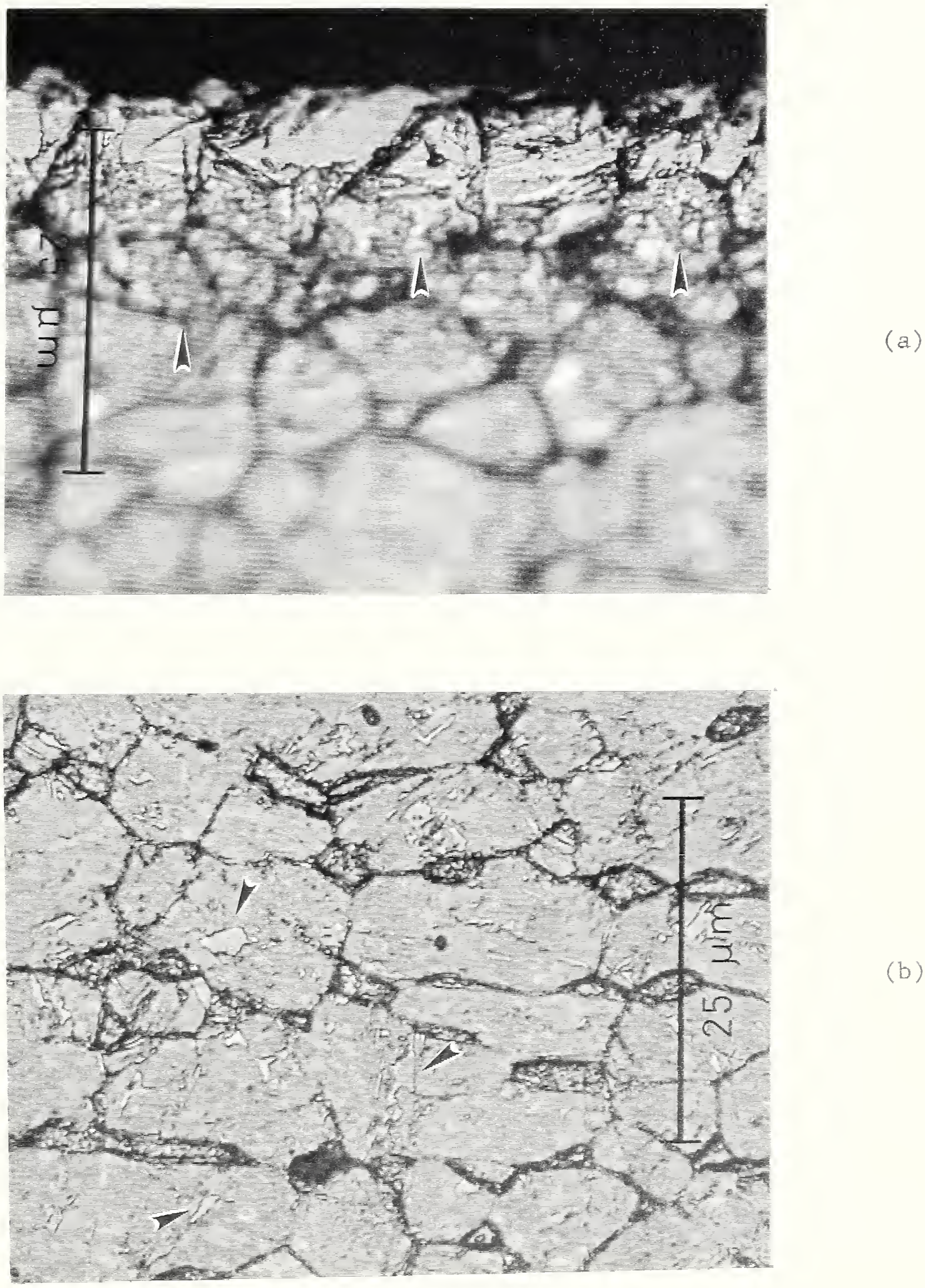


Figure 13. Photomicrographs of specimen NIST-Z1 taken near the surface (a), and center (b). The arrows show some of the retained austenite. Mag: Both X2000. Etch: Villela's

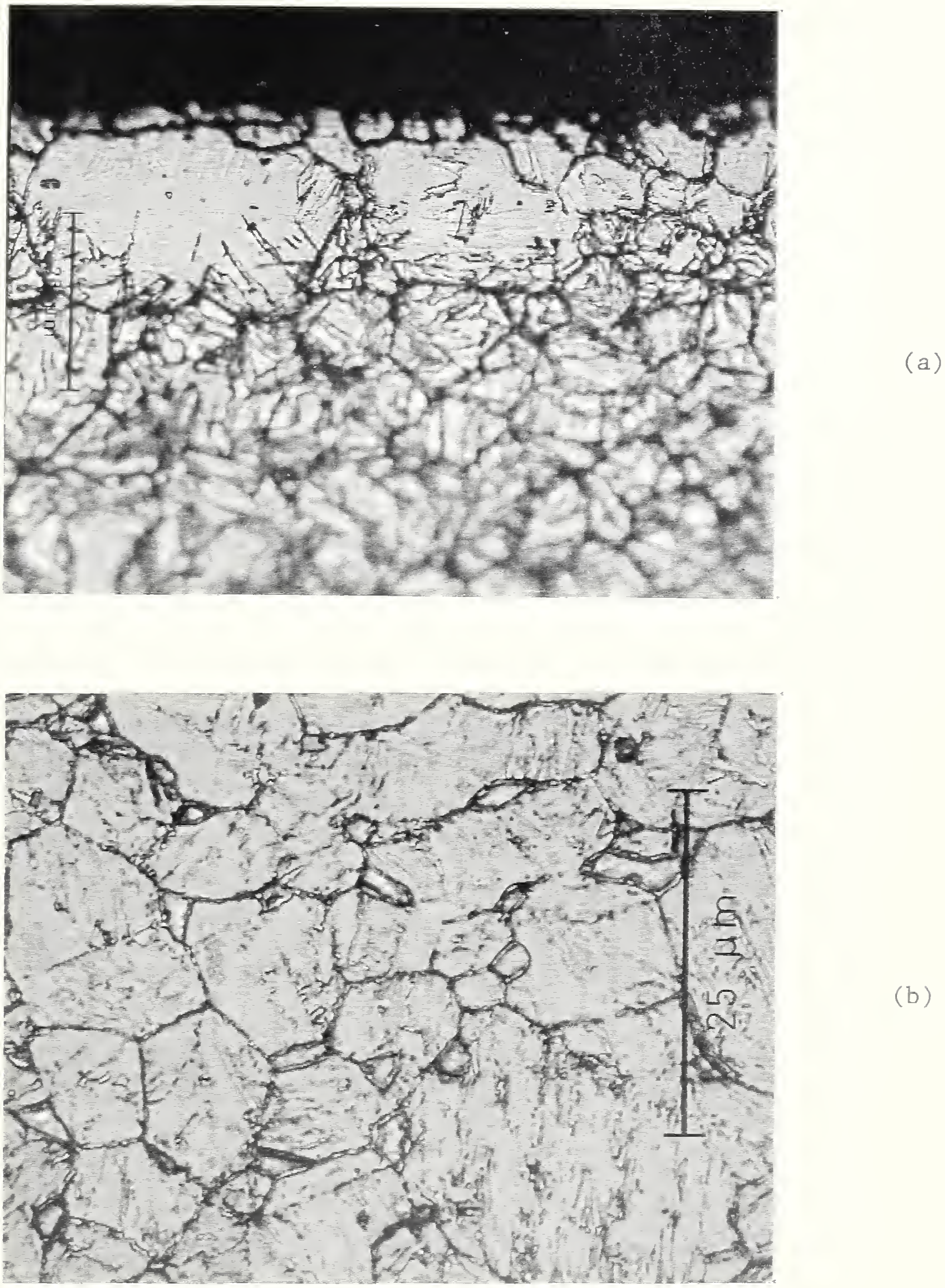


Figure 14. Photomicrographs of specimen NIST-Z2 taken near the surface (a), and center (b).
Mag: (a) X1000, (b) X2000. Etch: Villela's

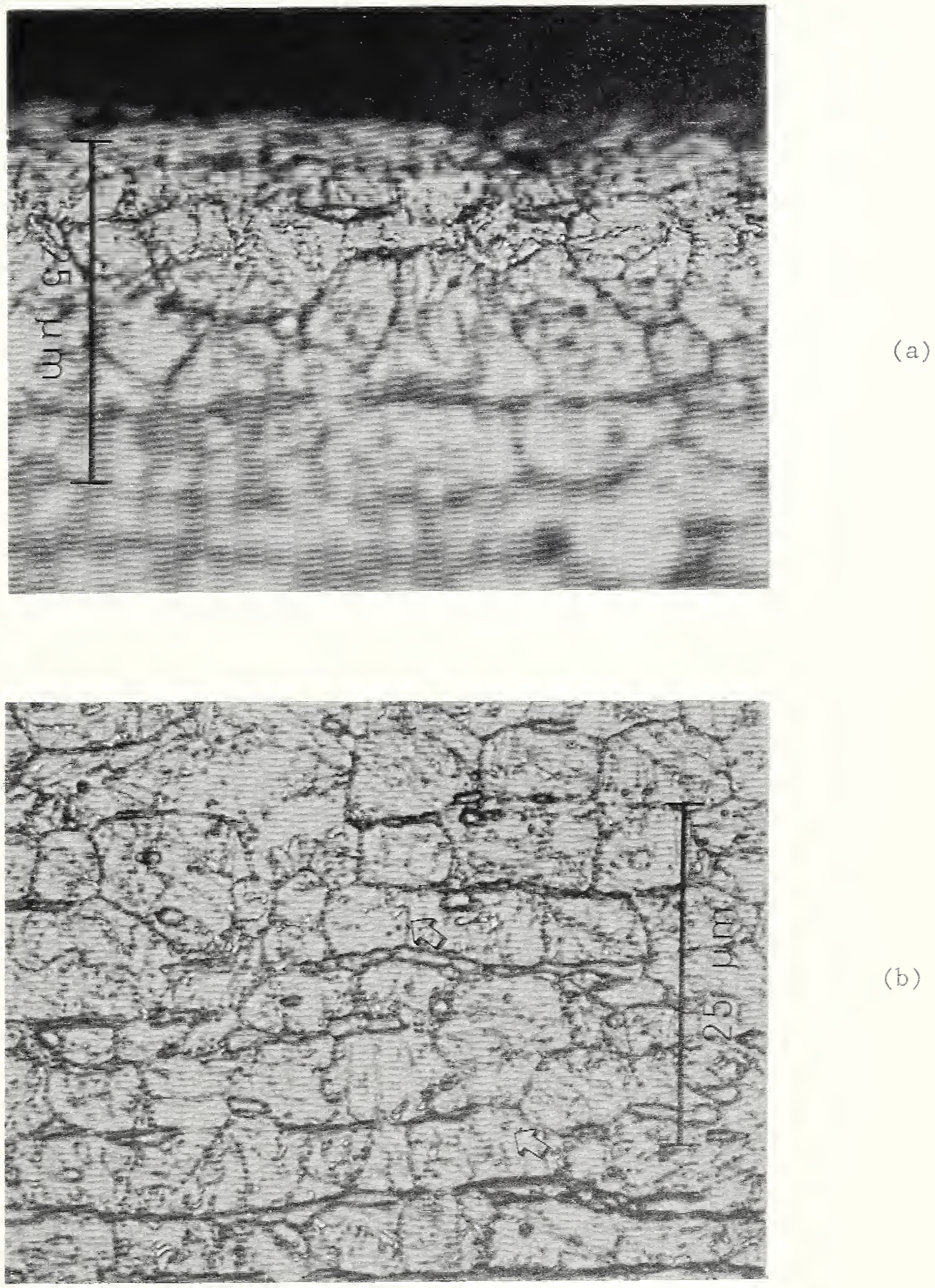


Figure 15. Photomicrographs of specimen NIST-Y taken near the surface (a), and center (b). There was no evidence of retained austenite on the surface, but some (arrows) was observed at the center. Mag: Both X2000. Etch: Villela's

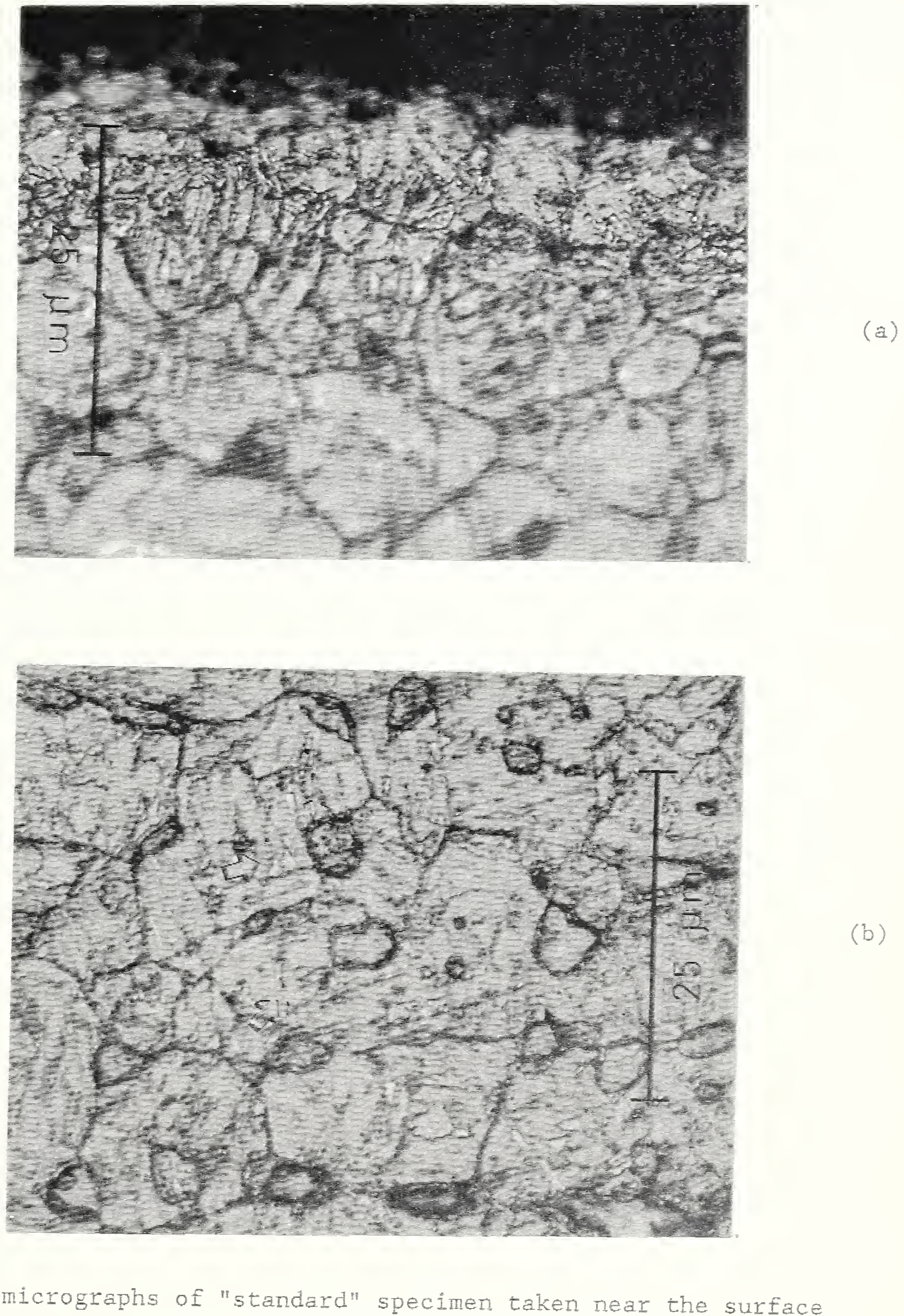


Figure 16. Photomicrographs of "standard" specimen taken near the surface (a), and center (b). There was no evidence of retained austenite on the surface, but some (arrows) was observed at the center. Mag: Both X2000. Etch: Villela's

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A "mishap" section, and other selected sections made from 17-7 precipitation hardening stainless steel were metallurgically examined, and it was concluded that the unusually large amount of retained austenite, greater than 7%, measured on the surface appears to have been produced during both the solution annealing and austenite conditioning steps of the heat treating process, but was revealed only after the austenite conditioning step. It is suspected that the excessive retained austenite occurred during the heat treating process where these sections may have been exposed to a furnace atmosphere that contained too much carbon or nitrogen. This exposure could have led to an increase in the amount of retained austenite both on the surface and internally.

12. KEY WORDS (6 TO 12 ENTRIES; ALPHABETICAL ORDER; CAPITALIZE ONLY PROPER NAMES; AND SEPARATE KEY WORDS BY SEMICOLONS)
atmosphere; carbon; microstructure; nitrogen; precipitation hardening; retained austenite; steel

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