Banding of MAR-Aging Steel

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MAR-aging steels have gained a respectable position among design engineers who demand ultra-high strength, reasonable ductility, and good fracture toughness, especially where outerspace and aerospace applications are concerned. This specialty steel category of MAR-aging alloys owes its unique properties to a complex hardening reaction, which involves precipitation of uniform intragranular ribbons (Ni_3Mo phase) on dislocations during the treatment cycle. Like any other metallurgical process, MAR-aging treatment cycles can become quite difficult, if not impossible. When that condition does exist, the end result is often called a dead heat by MAR-aging technologists. This paper examines a dead heat and makes appropriate comparisons to other live heats. Salient differences and interactive similarities are both studied in terms of microstructure, serial sections, chemical composition, and selected mechanical properties. The intent of this paper is to shed more light on the previously indistinct subject of banding.

Keywords alloy C-350, dead heat, microsegregation, banding, Ni-Mo rations, serial sections, spatial arrays, bandwidths

1. History

Clarence G. Bieber of the International Nickel Company (INCO) is credited with the discovery of MAR-aging steels.[1] Others at INCO's Paul D. Merica Lab in Suffren, NY, also excelled in the historic development of MAR-aging steel alloys; *viz.*, R.F. Decker, S. Floreen, T.W. Landig, C.J. Novak, J.T. Eash, A.J. Goldman, and E.P. Sadowski.[2–6] Additional contributions by Goldberg,[7] Pellisier,[8] and Khan[9] also merit mention. Over the years, technological advances for these MAR-aging steels have included many successful and innovative applications, some of which are mentioned in Table 1. More recently, and within the United States, meaningful applications have been attained by a team approach: end users working in close concert with two vital sources—the production mill (Teledyne Allvac, Richsburg, SC) and the primary distributor (Vasco Pacific, Montebello, CA).

Unfortunately, details about MAR-aging steel metallurgy are not very well understood by enough engineers and technologists. The technology transfer problem that exists between producers and consumers is the result of financial factors that have led to downsizing, mergers, and elimination of the research staffs that were assigned to this "minority market." of MAR-aging steels. As a result, contributions to the sum of total knowledge have been inadequate relative to the urgent needs and priorities of deserving consumer clients. Consequently, this publication is dedicated to a better understanding of a relevant topic; the homogeneity of a finished MAR-aging product in terms of its microscopic banding as an apparent result of an alloy segregation in the original ingot stage.

2. Materials

A single heat (JF25), which was known to have been a dead heat, was selected for this characterization study. A dead heat is one that will not respond to a corrective healing action (AR aging) for achieving an appropriate structure, property, or composition. By the same token, a live heat is defined as one that can be properly processed to attain optimum structure, property, and composition goals. This dead heat (JF25) was compared to two live heats bearing heat numbers 9552A and JD65. All three heats of alloy C-350 were produced by either Teledyne Vasco (PA) or Teledyne Allvac (SC). These three heats were each purchased by Vasco Pacific (CA) for qualification testing to support end usage.

Melting practice for these mill products involved state-of-the-art processes: induction vacuum melting (IVM) and consumable vacuum melting (CVM). IVM-CVM ingots were reduced to round bar at the producer mill using a proprietary reduction practice. Each round bar was ground smooth to eliminate surface oxides and seams from the fabrication cycle. Round bars were received in the mill annealed (MA) condition with a Rockwell C hardness number of HRC 33 to 35. All bars from all heats were processed to a similar diameter of about 15.87 mm (0.625 in). All samples were examined in a longitudinal plane of the rolling direction.

The nominal composition for MAR-aging steel (alloy C-350) is listed in Table 2. It is important to mention that all three heats of this investigation met the general requirements on nominal composition.

3. Methods

For this investigation, what is or is not classified as a dead or live heat was based upon both mechanical properties and microstructural observations.

Sample preparation for both photon and electron metallography used an ABRAPOL-2 (Struers Inc., Westlake, OH) machine where grinding was achieved by SiC papers and polishing involved diamond laps. The etchant used was one part Kallings reagent plus three parts ethanol.

Global (macroscopic) determinations of chemical composition were achieved by the methods of optical emission spectroscopy and wet chemistry. Local (microscopic) determinations of chemical composition were obtained using a scanning electron microscope (SEM) in the operating mode SEM:EDAX:EDS.

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Table 1 Applications of MAR-aging steel

Aircraft quill shafts	Belleville springs
Collets	Dimensional stability
Extrusion rams	Flexible hoses
Forging dies	Gas generator cases
Gimbal bearings	Gyroscopes
Helicopter rotors	Hopkinson bars
Hydrafoil struts	Landing gears
Load cells	Lunar roving vehicle
Mars Pathfinder	Nuclear components
Propeller shafts	Recoil springs
Rocket motor flexures	Trunnion pins

Table 2 Nominal composition of C-350 (wt.%)

Nickel	18.5
Cobalt	12
Molybdenum	4.8
Titanium	1.4
Aluminum	0.1
Silicon	0.1 max
Manganese	0.1 max
Carbon	0.03 max
Sulfur	0.01 max
Phosphorus	0.01 max
Zirconium	0.01
Boron	0.003

Quantitative metallography for bandwidths in terms of random serial scans and spatial distributions was applied and evaluated using appropriate statistical analyses. These numerous specimens and serial sections were examined relative to MAR-aging vs AR-aging processing parameters of both temperature and time. For alloy C-350, the MAR-aging approach uses mill annealing (MA) plus aging (A) as compared to the Ar-aging process of using solution annealed (SA) plus recrystallized (RX) plus aged (A). AR aging has been defined in the recent literature.[10]

Vickers microhardness tests (300 g, 15 s dwell time) were also applied to observe changes on the local level as a function of processing for the different metallurgical conditions and variables. Mechanical differences of the global order were examined by tensile tests.

4. Discussion and Results

Figures 1 and 2 are micrographs that define a live heat (9552A) versus a dead heat (JF25) for alloy C-350. Both illustrations are at an original magnification (M_0) of 125× with each specimen being in the etched and MA condition. Within a longitudinal plane of the rolling direction, the observations are at a random position. Here, a distinct difference is evident: heat 9552A is not banded, but heat JF25 is severely banded.

Table 3 reveals the symptoms of a dead heat (JF25) in terms of inadequate ductility (% El) and malleability (% RA). Notice that heat 9552A is not similarly effected. These mechanical properties were made on test samples, which had been age hardened by two independent testing laboratories, one in CA and another in TX.

Figures 3 and 4 show these elongation (El) and reduction in area (RA) differences in photomacrographs (M_0 = 7×) of rep-

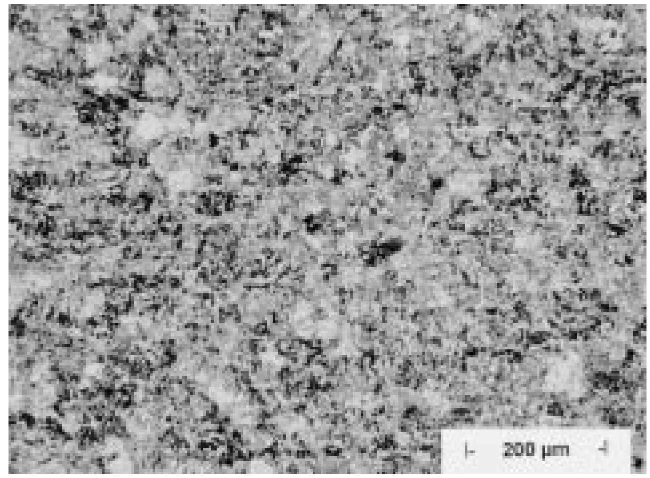

Fig. 1 MA C-350 (heat 9552A), 125×

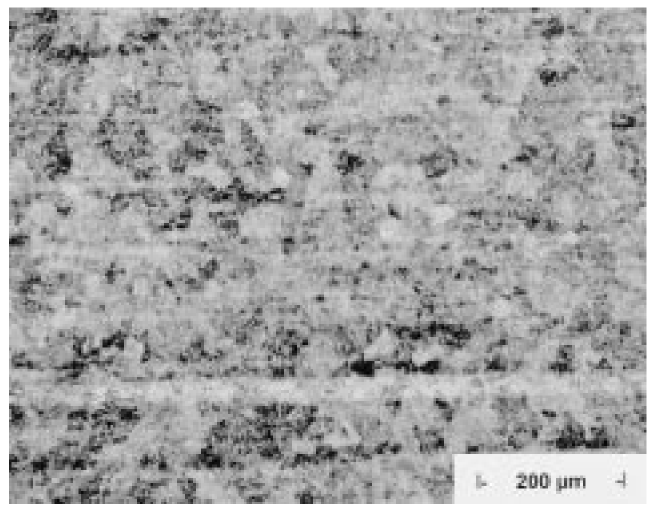

Fig. 2 MA C-350 (heat JF25), 125×

Table 3 Longitudinal tensile test results average

Test Result	Heat 9552A	Heat JF25
Ultimate	2399	2442
tensile strength, MPa (Psi)	(348,000)	(354,133)
Offset	2372	2351
yield strength, MPa (Psi)	(344,000)	(340,933)
Percent El	6	2
Percent RA	40	10

resentative tensile test bar samples, which have been broken and subsequently bonded together with plastic cement to demonstrate important volumetric factors. Figure 3 reveals the unbanded heat (9552A) with a normal test response where test bar volume is no longer constant and a ductile behavior prevails. Figure 4 illustrates the banded heat (JF25) with an abnormal test response where test bar volume is still almost constant and a more brittle behavior exists. Based on these illustrations (Fig. 1 to 4 and Table 3), heat JF25 is confirmed as being a dead heat.

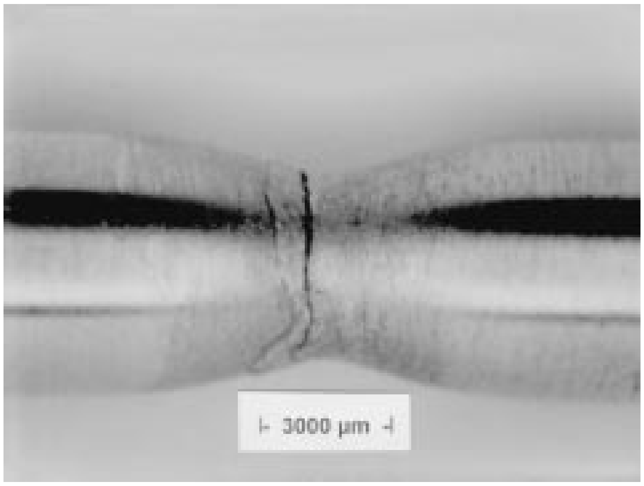

Fig. 3 Tensile bar of aged C-350 (heat 9552A), 7×

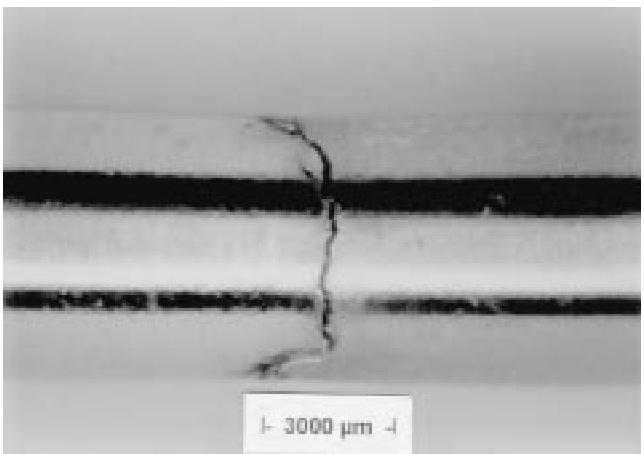

Fig. 4 Tensile bar of aged C-350 (heat JF25), 7×

Table 4 Heat treatment processing variables

Process	CA Source	TX Source
SA	1027 °C, 1 h, AC	1056 °C, 1 h, AC
Rx	927 °C, 1 h, AB 816 °C, 1 h, AB	789 °C, 1 h, WQ
A	512 °C, 4 h, AC 512 °C, 6 h, AC	517 °C, 1 h, AC

Notes: AC = air cool, AB = air blast, and WQ = water quench.

The etched and MA microstructure of the variety shown in Fig. 2 was confirmed in numerous other samples and within different serial sections for the same as-received condition of heat JF25. A logical concern posed this question: Are these bands of the mechanical or annealing type? Therefore, extensive heat treatment processing was mandated with this experimental objective: Is banding of MAR-aging steel caused by thermal or athermal events? Accordingly, appropriate heat treatments were executed by two unbiased laboratories in TX and CA.

Figure 5 documents a typical microstructure for a SA specimen of heat JF25 ($M_0 = 125\times$). The effect of SA processing is to dissolve the the bands such that solution annealing appar-

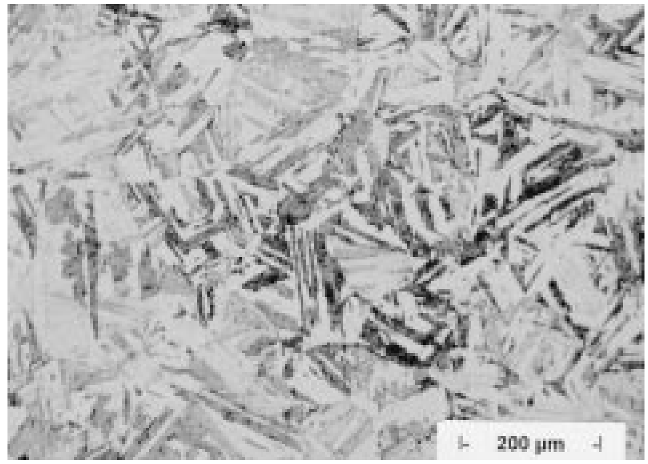

Fig. 5 SA C-350 (heate JF25), 125×

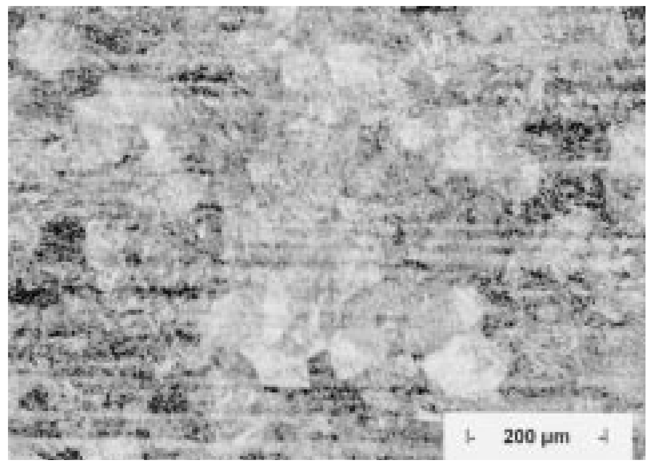

Fig. 6 RX C-350 (heat JF25), 125×

ently eliminates the problem of banding. Another important aftermath of a higher solution annealing temperature is to increase the average grain size that exists before recrystallization and aging are allowed to commence. This micrograph shows that the limit boundary temperature of 1093 °C has not been abused, because interstitial phases (as impurities) are not evident at either 125× or at higher magnifications, which were also examined. This observation confirms the work of both Pellissier[11] and Adair.[12] However, this observation does challenge the work of Goldberg,[7] who reported that banding could not be eliminated at a high temperature of 1200 °C for long treatment times. Goldberg's theory is probably based upon a different alloy composition.

Figure 6 documents an emblematic microstructure for heat JF25 in the RX state ($M_0 = 125\times$). The effect of different RX processing treatments, irrespective of time (t) and temperature (T) or sources, is to make the bands return for this dead heat (JF25). Thus, banding of heat JF25 cannot be eliminated by a low thermal cycle in the 815 °C range because of low diffusion rates, in agreement with Hall.[13]

Figure 7 is given to the subject of aging an RX condition to achieve optimized properties and structure.

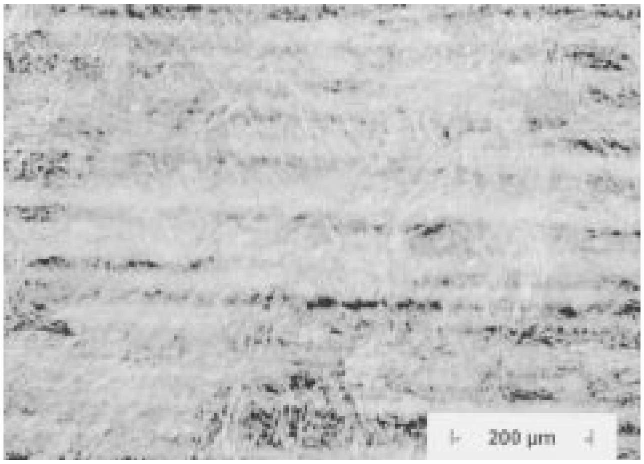

Fig. 7 AR-aged C-350 (heat JF25), 125×

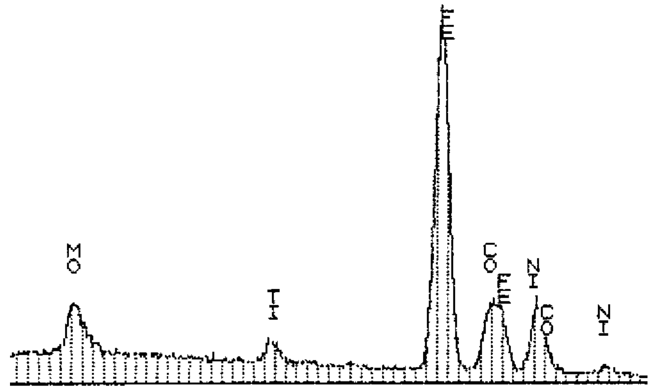

Fig. 8 SEM: EDAX: EDS scan C-350 (heat JF25)

After the combined events of AR aging (SA + RX + A), banding is quite conspicuous for all of the numerous specimens and serial sections that were examined. Figure 7 (M_0 = 125×) is considered to be representative of all such perusals for this age-hardened dead heat (JF25). Therefore, banding of MAR-aging steels is classified as an athermal event that is apparently restricted by limits of composition.

Figure 8 is a typical presentation (computergraph) where chemical composition was determined on a local (microscopic) basis. Here, semiquantitative test results are illustrated for a random sample in the MA condition. Figure 8 yielded these important test observations: the Ni-to-Mo ratio was improper at 3.94 and the molybdenum content was high at 5.55 wt.% All computergraphic scans (SEM:EDAX:EDS) for this dead heat (JF25) did reveal serious variations for both the Ni:Mo ratio and Mo compositions. For the live heats, results were not nearly as extreme. For example, typical Ni:Mo ratios were about 3.85 maximum and typical molybdenum contents were less than about 4.8.

Decker[5] reported that the amount of molybdenum is controlling. Decker stated that a 2% addition of Mo improves the toughness, a 2 to 5% addition age hardens by the precipitation of uniform intragranular ribbons of Ni_3Mo on dislocations, and additions greater than 5% aggravate banding. Decker defined aggravated banding as an undesired overabundance of the Ni_3Mo ribbon phase with an associated embrittlement effect. Campbell et al.[14] have argued that the molybdenum content needs to be controlled in the range of 4.6 to 5.2 wt.%. Accordingly, a more quantitative study of banding in MAR-aging C-350 steel was justified.

Figure 9 demonstrates typical spatial variations for the Mo content of this dead heat (JF25). On the basis of SEM:EDAX:EDS, Mo varies in the range of 3.01 to 5.59 wt.% as a function of specimen location in numerous serial sections.

Figure 10 shows typical bandwidths that exist as a function of both temperature and processing. In the MA condition, bandwidths vary from about 18 to 40 μm. Banding is almost nil in the SA condition, because very high diffusion rates have caused annihilation of the dislocation oriented Ni_3Mo phase ribbons; *i.e.*, annealing has created a proper solutionizing of all alloy hardening elements including Co, Mo, and Ti. Bandwidths for the SA plus RX condition are small and in the range of about 18 to 32 μm. Larger bandwidths of about 32 to 90 μm exist after a complete thermal processing cycle: A plus R plus A, which defines the AR-aging heat treatment method.

Figure 11 indicates that bandwidths undergo a coarsening-like event as a function of time-dependent factors. Overaging at 4 to 6.5 h causes the largest bandwidths to exist. Proper aging for only 1 h yields smaller bandwidths; therefore, this bandgrowth from small to large is described as a coarsening event. And, this is logical because the solid-state precipitation of Ni_3Mo ribbons acts to control both mechanical strength and metallurgical properties for this alloy.

Figure 12 and 13 (M_0 = 125×) show the extremes of bandwidth vs time at temperature during the aging process. Figure 12 is a typical photomicrograph of a test specimen, which received a 1 h aging treatment; a refined band width is here shown. Figure 13 shows a coarsened bandwidth for another specimen that was aged for a longer time (6.5 h) at temperature. If the molybdenum content exceeds about 5.2% (5.55 was here observed), it is evident that overaging causes coarsening of the band widths.

5. Conclusions

MAR-aging steels are very structure sensitive and quite responsive to minuscule changes in alloy content, especially with respect to the amount of molybdenum. MAR-aging steels are purchased in the MA condition with a material certification that says, if properly heat treated, optimum properties can be achieved. For live heats, this is an adequate approach, but in the case of dead heats, it is inadequate. For an application that depends on stiffness or tensile toughness, alloy segregation from the ingot stage cannot be tolerated in the final product. Therefore, these supplementary requirements (SR) for the associated purchase document (MIL-S-46850D) are mandated by the findings of this investigation.

SR(1): Metallography of longitudinal specimens in the MA condition shall be required. If banding is detected (as in Fig. 2), additional tests shall be required.

SR(2): Tensile tests of an aged sample shall be required as an acceptance standard. If EI and RA are significantly low (as in

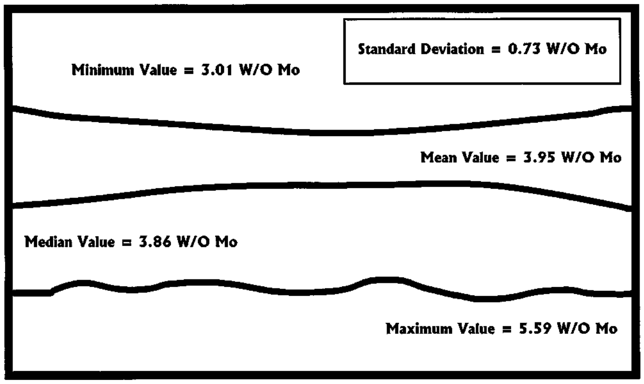

Fig. 9 Variation of Mo content (heat JF25)

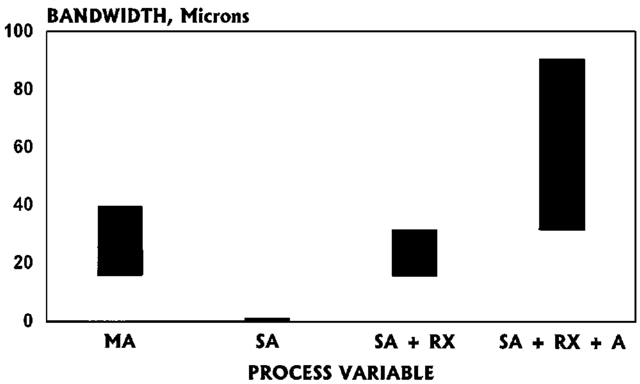

Fig. 10 Temperature effect on bandwidth (heat JF25)

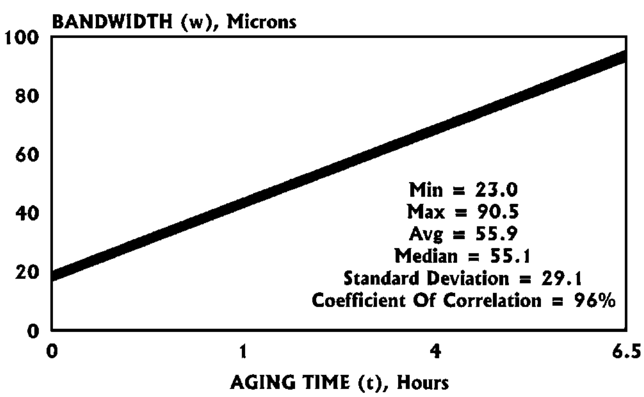

Fig. 11 Time effect on bandwidth (heat JF25)

Fig. 4), a dead heat exists and the associated Lot shall be deemed as a valid rejection.

SR (3): Molybdenum amounts in alloy C-350 shall be restricted to a control range of 4.9+/–0.3 wt.% Mo (as in Ref 14). The absolute limit for these alloys shall be 5.2 wt.% Mo, irrespective of the analytical method that is used to determine either global or local composition.

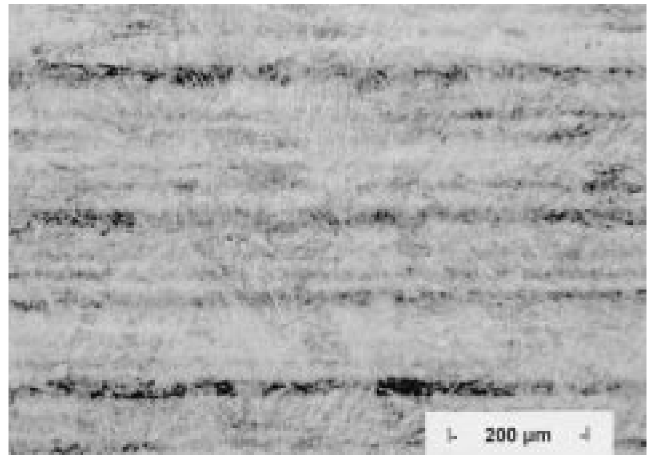

Fig. 12 Banding after 1 h aging (heat JF25), 125×

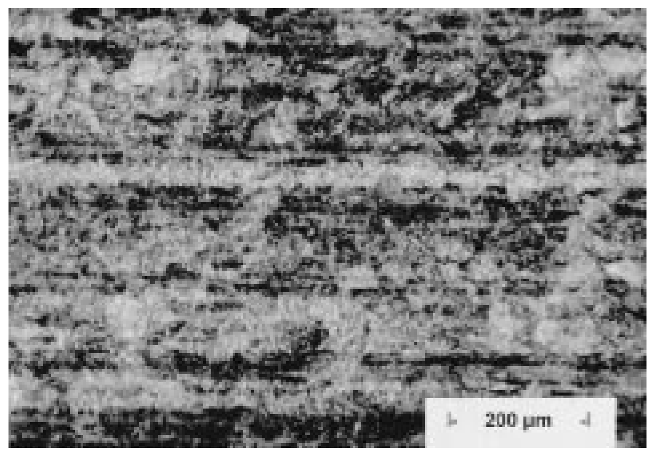

Fig. 13 Banding after 6.5 h aging (heat JF25), 125×

SR (4): Retests to challenge a specific rejection activity shall use AR-aging heat treatment techniques (as in Ref 10). Overaging to cause coarsening of microsegregated bandwidths shall not be applied (as in Fig. 13).

In this manner, dead heats can be properly separated from live heats at the beginning of a given design project. Thus, potential injury and subsequent damage claims can be circumvented by better quality control methods and improved purchase requirements.

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