EFFECT OF TMP VARIABLES UPON STRUCTURE AND PROPERTIES IN ODS ALLOY HDA 8077 SHEET

M. F. Rothman
H. M. Tawancy
STELLITE DIVISION, Cabot Corp., Kokomo, Indiana

The effects of oxide content level and variations in thermomechanical processing upon the final structure and properties of HDA 8077 sheet have been systematically examined. It was found that creep strength and formability are substantially influenced by both oxide content and TMP schedule. Variations in creep properties obtained appear to correlate with observed microstructures.

INTRODUCTION

As the need for increasing gas turbine engine operating temperatures has continued throughout the past decade, engine designers in recent years have been turning their attention more and more to the potential application of oxide dispersion strengthened (ODS) alloys in advanced engine designs. This is particularly true in the case of sheet materials for use in the combustor sections of gas turbines.

HAYNES* Developmental alloy No. 8077 is a nickel-base ODS alloy containing nominally 16% chromium, 4% aluminum, and up to 2 weight percent yttrium oxide. Produced by thermomechanical processing (TMP) of mechanically alloyed powder, HDA 8077 sheet is a candidate for use in advanced combustor designs by virtue of its high creep strength and good oxidation resistance in comparison to conventional alloys at temperatures exceeding 1255°K (1800°F).

The final structure and properties of ODS alloy sheet materials such as HDA 8077 can depend largely upon the details

* HAYNES is a registered trademark of Cabot Corporation.

of the TMP techniques used in sheet production. In the present study, the objective was to determine the structure/property response of HDA 8077 sheet to variations in TMP schedule, while simultaneously examining the differences in response as a function of alloy oxide content. In terms of criticality to the application in question, the properties of interest were creep strength and formability.

EXPERIMENTAL PROCEDURE

Two 41 Kg (90 pounds) master lots of HDA 8077 powder were prepared using mechanical alloying techniques. The first contained 1.33 weight percent yttrium oxide, and the second 0.80 weight percent. Powder lots of about 3 Kg (6.6 pounds) were canned in evacuated steel containers and press-forge consolidated. Each lot was then hot cross rolled in the original steel container to produce plate approximately 6.4 mm (0.250 inch) thick. This plate was then sectioned, re-canned, and finish hot cross rolled to a final gauge of 1.3 mm (0.050 inch).

After final decanning, samples were cut from each lot of sheet and subjected to grain growth annealing treatments of from 1505°K (2250°F) to 1616°K (2450°F) for 30 minutes. This was done to determine the minimum temperature required to produce the large, abnormally grown grains necessary for high creep strength. Based upon subsequent microstructural evaluation, the remainder of each hot rolled sheet was given an appropriate anneal. All sheets were then sand blasted and pickled to remove surface oxide scale.

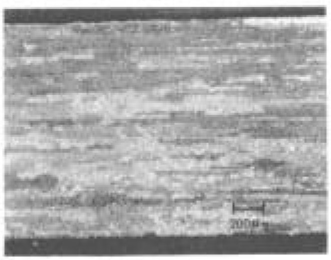
The various TMP parameters evaluated in the study are outlined in Table 1. Combinations were selected identically for both the high oxide content and low oxide content powders.

Table 1. TMP Parameters Evaluated.

Parameter	Variations
Powder Consolidation Temperature	1311°K, 1450°K
Hot Rolling Temperature	1241°K, 1311°K, 1380°K
Finish Rolling Reduction/Pass	20%, 30%

RESULTS

The effect of annealing temperature upon the microstructure of the sheet produced by the different TMP routes examined in the study is illustrated in Figures 1 and 2. It is apparent from Figure 1 that for material hot rolled at the lower temperatures, annealing at temperatures below 1616°K (2450°F) does produce some degree of abnormal grain growth; however, large areas of the fine grains which characterize the as hot rolled structure are still in evidence.



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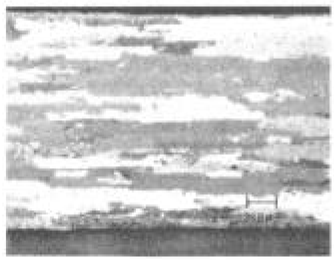

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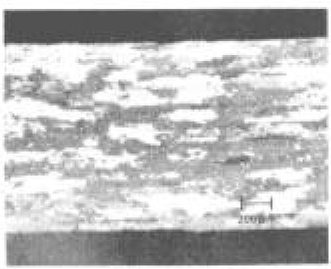

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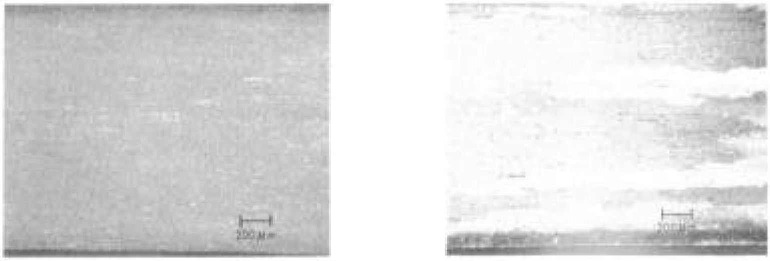
FIGURE 1: Structures obtained by annealing sheet produced from 0.80% oxide content powder at various temperatures. All material taken from powder consolidated at 1311°K and hot rolled at 1261°K using 20% reductions per pass. Sheet annealed for 30 minutes. (a) 1505°K; (b) 1561°K; (c) 1616°K.

With the material hot rolled at the higher temperatures, the effect of annealing temperature is even more pronounced. Figure 2 shows the complete lack of response to annealing for 30 minutes at 1533°K (2300°F) for sheet rolled at 1380°K (2025°F).

In the light of these results, all materials destined for further evaluation were annealed at 1616°K (2450°F) for 30 minutes.

The various optical microstructures produced as a function of the prior thermomechanical history are described in Table 2, along with the typical macrostructural grain sizes observed upon macroetching the surface of the sheets. It was noted that all of the sheets produced from the 1.33% oxide content

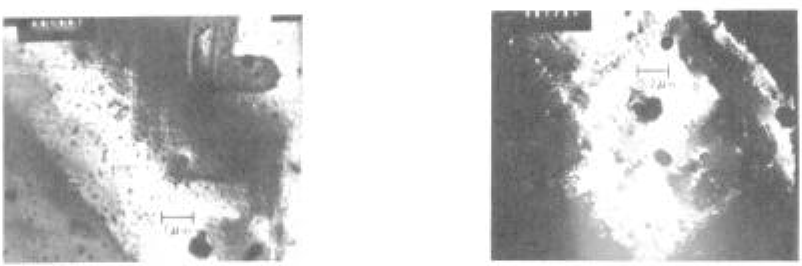


(a) (b)

FIGURE 2: Structures obtained by annealing sheet produced from 0.80% oxide content powder at various temperatures. All material taken from powder consolidated at 1450°K and hot rolled at 1380°K using 20% reductions per pass. Sheet annealed for 30 minutes. (a) 1533°K; (b) 1616°K.

powder had microstructures which exhibited oxide banding. This was not observed in the 0.80% oxide content material. As may be seen from the data in Table 2, the higher oxide content material also exhibited an apparent tendency towards a finer macrostructural grain size than that for the lower oxide content sheet.

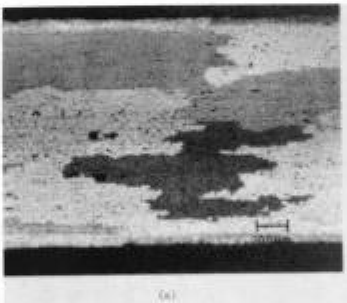
Typical transmission electron micrographs of both high and low oxide content material in the recrystallized condition are shown in Figure 3. The rectangular dislocation cell structure shown in Figure 3 was present at both oxide levels, with the extent dependent upon thermomechanical processing history; however, the cell size in the high oxide content material was generally much smaller than that for the lower oxide content material. In most cases, the structure of both materials was characterized by a mixture of this cell structure and three dimensional forests of random dislocation tangles.

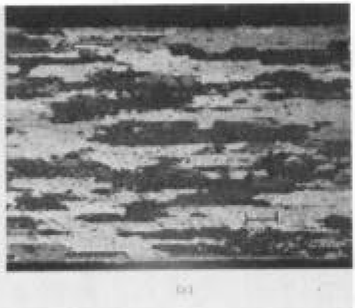


(a) (b)

FIGURE 3: Transmission electron micrographs of sheet material produced by consolidating powder at 1450°K and hot rolling at 1380°K using 20% reductions per pass. Sheet annealed for 30 minutes at 1616°K. (a) 0.80% oxide content powder; (b) 1.11% oxide content powder.

The variation in optical microstructure observed as a function of changing the rolling temperature is illustrated in Figure 4. As the rolling temperature was decreased from





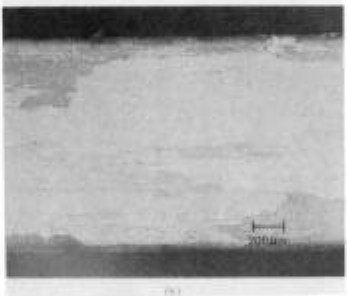


FIGURE 4: Structures obtained by finish rolling sheet produced from 0.90% oxide content powder at various temperatures. All material taken from powder consolidated at 1311°K and hot rolled using 20% reductions per pass. Sheet annealed for 30 minutes at 1616°K. (a) 1380°K; (b) 1311°K; (c) 1241°K.

1380°K (2025°F) to 1241°K (1775°F), the typical number of the grains through the thickness of the sheet increased. This was accompanied by a reduction in the macrostructural grain size, as shown in Table 2.

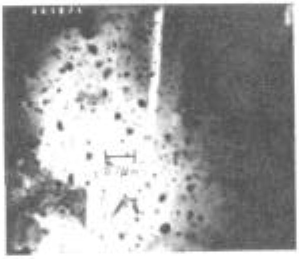
The effect upon dislocation substructure is illustrated by the transmission electron micrographs presented in Figure 5. As the finish rolling temperature decreased from 1380°K to 1311°K, the size of cells formed by pinned dislocations appeared to generally increase. The overall effect upon the relative quantities of cell structure and random dislocation tangles present has not yet been established; however, it was found that when the finish rolling temperature was reduced to 1241°K, the cell structure was largely absent.

The resulting effect upon the optical microstructure of increasing the reduction per pass during finish rolling from 20% to 30% is shown in Figure 6. The number of grains through the thickness of the sheet was markedly increased, while the macrostructural grain size, as related by the data in Table 2, was little affected.

Considering the degree of variation in sheet structure observed as a function of thermomechanical history, a substantial spread in creep strength and formability properties was

Table 2. Structures Produced as a Function of Prior TMP History.

Powder Consolidation Temperature (°K)	Rolling Temperature (°K)	Finish Rolling Reduction Per Pass (%)	Typical Macrostructural Grain Diameter (mm)	Minimum No. of Grains Through Thickness	Type of Grains
			0.80% Oxide Powder		
1311	1241	20	1.6 to 3.2	8	Pancake
1311	1241	30	3.2	10	Low Ratio Pancake + Fines
1311	1311	20	3.2 to 6.4	4	Pancake
1311	1311	30	3.2 to 6.4	8	Pancake + Fines
1311	1380	20	9.5 to 12.7	6	Pancake
1450	1380	20	9.5	6	Pancake + Fines
			1.33% Oxide Powder		
1311	1241	20	1.6	10	Pancake
1311	1241	30	1.6	15	Pancake + Fines
1311	1311	20	1.6 to 3.2	6	Pancake + Fines
1311	1311	30	1.6 to 3.2	10	Pancake + Fines
1311	1380	20	12.7	3	Pancake + Fines
1450	1380	20	6.4 to 9.5	6	Pancake



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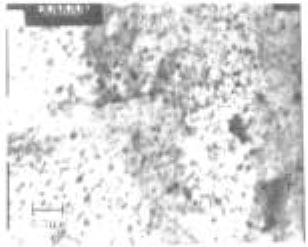

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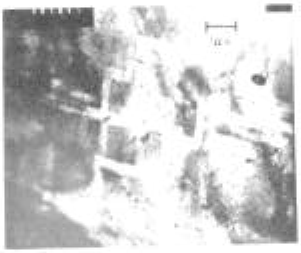

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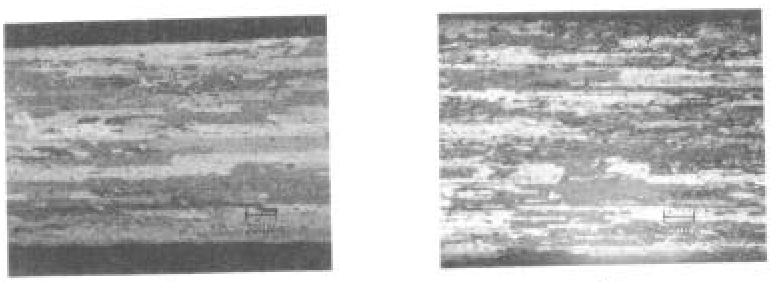
FIGURE 5: Transmission electron micrographs of sheet obtained by finish rolling 0.80% oxide content material at various temperatures. All material taken from powder consolidated at 1311°K and hot rolled using 10% reductions per pass. Sheet annealed for 30 minutes at 1616°K. (a) 1380°K; (b) 1311°K; (c) 1241°K.

anticipated. Such a spread was observed, as related by the Olsen Cup Test and 1255°K (1800°F) creep rupture test properties presented in Table 3.



(a) (b)

FIGURE 6: Structures obtained by finish rolling sheet produced from 1.33% oxide content powder using different reductions per pass. All material taken from powder consolidated at 1311°K and hot rolled at 1241°K. Sheet annealed for 30 minutes at 1616°K. (a) 20% reduction/pass; (b) 30% reduction/pass.

It is apparent from the data in Table 3 that, as a class, the 0.80% oxide material was stronger in creep than the 1.33% oxide material, while the formability of the sheets was comparable. At both oxide levels, formability was seen to be generally reduced as finish rolling temperature decreased, but was affected only slightly by the size of the finish rolling reduc-

Table 3. Properties Produced as a Function of Prior TMP History.

Powder Consolidation Temperature (°K)	Rolling Temperature (°K)	Finish Rolling Reduction Per Pass (%)	Stress (MPa)	Avg.* Life (Hrs)	Lowest Life (Hrs)	Final Creep Reading (%)	Rupture El. (%)	Average** Olsen Cup Depth (mm)
0.80% Oxide Powder								
1311	1241	20	83	29	21	1.7	2.6	3.5
1311	1241	30	--	--	--	--	--	3.6
1311	1311	20	97	37	6	0.4	2.4	5.2
1311	1311	30	--	--	--	--	--	4.5
1311	1380	20	90	70	7	0.3	2.2	7.0
1450	1380	20	90	253	155	0.5	4.1	7.6
1.33% Oxide Powder								
1311	1241	20	83	121	82	0.4	1.7	3.5
1311	1241	30	--	--	--	--	--	3.7
1311	1311	20	83	77	40	0.5	1.8	3.9
1311	1311	30	83	90	71	0.4	1.8	3.4
1311	1380	20	83	51	25	0.5	1.6	6.9
1450	1380	20	83	114	7	0.5	2.5	7.9

* Logarithmically averaged ** Minimum of 3 tests

tion per pass. Consolidating powder at the higher temperature appeared to improve the formability of the resulting final sheet product in both cases.

As for TMP effects upon creep properties, it was interesting to observe opposite trends for the two oxide content levels. In the low oxide content material, creep strength tended to increase with increasing rolling temperature, and to be most consistent when the higher powder consolidation temperature was employed. In the high oxide content material, creep strength tended to decrease with increasing rolling temperature, and to be most consistent when the lower powder consolidation temperature was used. Based upon limited testing, increased reductions during finish rolling appear to have only a minor beneficial effect upon creep life.

In all, the best combination of formability, creep strength and creep strength consistency appeared to be obtained with the low oxide content material, forge consolidated and rolled at the higher temperatures.

DISCUSSION

It is apparent from the results of this study that the relationship between the structure and properties in HDA 8077 sheet is complex. Examining the data presented in Tables 2 and 3 for the low oxide content material, it is observed that the grain size increased when the rolling temperature was increased from 1311°K to 1380°K. The increase in creep strength, which might have been expected to accompany the larger grain size, was not seen, however.

From the structures illustrated in Figure 5, it is known that the size of rectangular dislocation cell structure decreased when the rolling temperature was increased from 1311°K to 1380°K. These dislocation cell walls, or low angle grain boundaries, could act as sources of dislocations which migrate to the high angle grain boundaries and facilitate grain boundary sliding. In such a case, decreasing the relative cell size would be expected to increase the efficiency of this process, and possibly reduce creep strength even though the grain size is increased. Of course, changing the relative amounts of cell structure and random tangles present would also affect this process, but the effect of rolling temperature on this balance has not yet been fully established.

This rationale cannot be used to explain the lower creep strength exhibited by the low oxide content material rolled at

1241°K, wherein the dislocation cell structure was found to be largely lacking. In view of the massive dislocation tangles observed in this material, it could be expected that, despite the finer grain size, grain boundary sliding would be inhibited, and a higher creep strength observed. This seems to be confirmed by the lack of room temperature formability exhibited by the material inspite of the transgranular nature of the cup test failures. This is indicative of a lack of dislocation mobility.

The answer could be that there is a second major source of dislocations, active only at elevated temperature. The oxide particle/matrix interface could be such a source. Some possible evidence for dislocations emanating from the larger particle/matrix boundaries has been obtained in this study; however, the data are far from conclusive.

The effect of increasing the number of oxide particles on such a mechanism is unclear. On the one hand, the increased number of particle/matrix interfaces would increase the number of dislocation sources. On the other hand, the increased number of particles would tend to inhibit the migration of emanating dislocations to the high angle grain boundaries. This could account for the increase in creep strength observed for the high oxide material over the low oxide material at the lower rolling temperature, where the cell substructure is largely absent. For the high oxide content material produced at the higher rolling temperature, the resulting finer dislocation cell structure could be expected to account for the lower creep strength relative to both the material rolled at 1241°K, and also to the lower oxide content material.

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