## **Recrystallization Mechanisms In A Previously Cold – Rolled Duplex Stainless Steel**

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### Abstract

Investigation has been carried out on the recrystallization mechanisms of three structural regimes: ferrite, reversed austenite and untransformed austenite. A duplex stainless steel alloy, Fe-21% Cr-7% Ni and the  $1050^{\circ}$ C tie-line single-phase austenitic and ferritic alloys were used. The alloys were cold rolled at  $-70^{\circ}$ C and  $-196^{\circ}$ C to obtain martensite and subsequently annealed for 3 minutes at different temperatures in the  $500 - 900^{\circ}$ C range. The ferrite and reversed austenite were found to recrystallize by the continuous (sub-grain coalescence) mechanism. The untransformed austenite was found to recover with persistent banded morphology suggesting the discontinuous (grain-boundary migration) mechanism or the formation of new recrystallization grains at the intersections.

## 1.0 Introduction

Fine grained metals and alloys have received considerable attention because of enhanced mechanical properties. Refinement is obtained by the process of recrystallization, a process which often involves cold deformation and subsequent annealing. The residual stress associated with deformation which limits the practical application of structural metals is released during the annealing process. However the final microstructure and the associated mechanical properties depend on the mechanisms involved in the heat-treatment.

Two recognised recrystallization mechanisms are continuous (sub-grain coalescence) and discontinuous (grain boundary migration) mechanisms [1-3]. During recrystallization dislocation-poor grains grow replacing the deformed dislocation-rich matrix. The mobility of the grain boundaries (low in the case of continuous and high for discontinuous mechanism) is described by the parameter  $K^1$ . Specifically

 $K^{1} = 2\sigma_{b}M = 2\sigma_{b}\frac{D_{o}}{NkT}\exp\left(-\frac{Q}{RT}\right) \quad [4] - - - - - (1)$ 

where M = grain boundary mobility (velocity per unit driving force),  $\sigma_b$  = energy of grain boundary, D<sub>o</sub> = normal preexponential term for atomic migration, N = number of atoms on unit area of boundary, Q = the measured activation energy.

However the recrystallization mechanisms have remained a subject of debate among materials scientists [5, 6, 7]. Stable recrystallization grains have been found to form by mechanism(s) other than continuous or discontinues.

Specifically Andrey Belyakov *et al* [8] worked on an Fe-27% Cr-9% Ni dual-phase (ferrite-austenite) stainless steel and identified continuous and early discontinuous recrystallization in ferrite ( $\alpha$ ) and austenite ( $\gamma$ ) respectively. The alloy was severely deformed but the authors did not report any martensite formation.

In this work deformation of a duplex alloy will be done at sub-zero temperatures to ensure martensitic reaction. This will result, during subsequent annealing, in three structural regimes: untransformed austenite, reversed austenite and ferrite. The recrystallization mechanisms in these regimes would be of interest.

### 2.0 Materials and method

A duplex stainless steel (U50D) and the 1050°C tie-line single phase, austenitic (U50A) and ferritic (U50F) alloys were used.

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The composition of the U50D alloy is shown in Table 1. Table 1: Composition of the U50D alloy

С	Cr	Ni	Mn	Si	Мо	Cu	S	р
0.026	20.60	6.62	1.60	0.46	2.45	1.64	0.018	0.031

The alloys were solutionised at 1050°C, 1030°C and 1300°C respectively for 1 hour and water-quenched. This was followed by cold-rolling at -70°C and -196°C to 20 and 80% reduction.

Subsequently the specimens were annealed at temperatures in the  $500 - 900^{\circ}$ C range for 3 minutes and water-quenched. Structural studies were carried out on the Transmission Electron Microscope.

# 3.0 Results and Discussion

After annealing a previously 80% deformed U50D for 3 minutes at 700°C the  $\alpha$ -phase is seen to recover with sub-grain formation, plate 1.



Plate 1: U50D deformed 80% at -196°C and annealed at 700°C. Bright Field (BF) image shows development of sub-grains in  $\alpha$ 



Plate 2: U50F deformed 20% at -196°C. Bright Field (BF) electron micrograph reveals a cell-structural appearance of the dislocations.

In view of the fact that in the as-deformed condition the dislocations in the  $\alpha$ -phase exhibit a cell-structural appearance (plate 2).

The sub-grains are assumed to have been inherited from the deformed structure by rearrangement of the dislocations during recovery; infact the size of the sub-grains, is similar to the deformation cells. The sub-grain, being of low-angle boundary configuration will have low mobility. Subsequent formation of stable recrystallization nuclei and growth in the  $\alpha$ -phase would therefore involve the coalescence of the sub-grains by the annihilation of the sub-boundaries in agreement with [8]. Indeed, evidence for this is illustrated in plate 3.



Plate 3: U50D deformed 80% at -196°C and annealed at 800°C for 3 minutes.  $\alpha$  has begun to recrystallize and subboundaries (arrowed) are disappearing

The sub-boundaries for sub-boundaries are seen to contain more dislocations than there are within the grains and within the larger grains disappearing sub-boundaries (arrowed) are seen. The reversed  $\gamma$  also recrystallizes via sub-grain formation, plate 4.



Plate 4: U50D deformed 80% at -196°C and annealed for 3 minutes at 800°C showing sub-grains and recrystallization in and  $\alpha$  respectively



Plate 5: High Resolution Dark Field (HRDF): U50D deformed 80% at -196°C, showing thin inter-lath untransformed austenite reversed  $\gamma$ 

It is of note that this plate is for the alloy previously deformed to 80% reduction, a condition which leaves very little  $\gamma$  untransformed, as indicated in plate 5.

Further evidence is provided in plate 6 in which the dislocations within the martensite ( $\alpha^{1}$ ) laths are inhormogeneously distributed, appearing to have high and low density across the length; this approximates to a cell structure within the laths.



Plate 6: High Resolution Dark Field (HRDF): U50D deformed 20% at -196°C. Dark regions is untransformed γ. The martensite laths appear to exhibit cell-structural appearance of dislocation.



Plate 7: U50D deformed 20% at -196°C followed by 3 minutes at  $900^{\circ}$ C. Recrystallized  $\gamma$  has developed annealing twins

Reversed  $\gamma$  would inherit this structure. Subsequently recrystallization would proceed by sub-boundary coalescence. An indirect evidence of this mechanism is provided by the reversed  $\gamma$  recrystallizing with annealing twins.

Nucleation of recrystallization is favoured energetically by the coalescence of sub-boundaries (resulting in the formation of twins) on the basis of the inequality:

 $\sum S_{cb} \sigma_{cb} + \sum S_{nb} \sigma_{nb} < S_{gb} \sigma_{gb} \quad [9] \qquad - \qquad - \qquad (2)$ 

where  $\sum S_{cb}$  and  $\sum S_{nb}$  are the total area of coherent and non-coherent twin boundaries respectively,  $\sigma_{cb}$  and  $\sigma_{nb}$  are the specific energy of coherent and non-coherent twin boundaries respectively.  $S_{gb}$  and  $\sigma_{gb}$  are similarly defined for the original sub-grain boundaries.

For the untransformed  $\gamma$ , the recrystallization mechanism may be different from the sub-boundary coalescence discussed for  $\alpha$  and reversed  $\gamma$ . For this regime of  $\gamma$  sub-boundary migration mechanism is indicated. The difference shows even at the early stages. Referring to plate 8, after 3 minutes annealing at 700°C of 80% deformed U50A sample, the untransformed  $\gamma$  has undergone a considerable degree of recovery but a banded structure persists which is related to the deformed state.



Plate 8: U50A deformed 20% at -196°C and annealed at 700°C: Bright Field (BF) image of untransformed  $\gamma$  revealing extensive recovery and persistent deformation-induced banded morphology

These bands have been referred to severally as pre-formed recrystallization nuclei, or transition bands, and the intersections as sites for recrystallization.

The recrystallization of deformed untransformed  $\gamma$  (with banded structure in the deformed state) would be associated with high-angle boundary migration [4, 10, 11].

Whether the untransformed  $\gamma$  recrystallizes by the bulge mechanism or the formation of new recrystallization grains at the transition boundaries, the mechanism is different from the sub-boundary coalescence mechanism described for  $\alpha$  and reversed  $\gamma$ .

### 4.0 Conclusion

During the isothermal annealing of Duplex stainless steel previously deformed to obtain martensite three structural regimes occur: ferrite, reversed austenite and untransformed austenite,

The ferrite and reversed austenite recrystallize by the continuous (sub-grain coalescence) mechanism. This involves formation of sub-grains and subsequent disappearance (or annihilation) of the sub-boundaries.

The untransformed austenite recovers with persistent band structure. This would lead to the continuous (grain-boundary migration) mechanism of recrystallization or formation of new recrystallization grains.

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