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**DISLOCATION SUBSTRUCTURES IN
HIGH-ENERGY-RATE-FORGED AND
PRESS-FORMED 21-6-9 STAINLESS STEEL**

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SUBJECT DESCRIPTORS

**Stainless Steel
Forging
Dislocation Structures
Deformation**

**ROCKWELL INTERNATIONAL
ATOMICS INTERNATIONAL DIVISION
ROCKY FLATS PLANT
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DISLOCATION SUBSTRUCTURES IN HIGH-ENERGY-RATE-FORGED AND PRESS-FORMED 21-6-9 STAINLESS STEEL

*Elane C. Sanderson, Arvel W. Brewer, Robert W. Krenzer, and George Krauss**

Abstract. A Transmission Electron Microscope (TEM) was used to establish that the substructure of Press Formed (PF) 21-6-9 stainless steel toroids was characterized by a dislocation cell size finer, about $0.16\ \mu\text{m}$, than that of about $0.29\ \mu\text{m}$, of High-Energy-Rate-Formed (HERF) processed toroids. In addition, HERF processed material showed areas of a coarser, well-developed subgrain structure characteristic of hot work deformation. The formation of the substructures, as-well-as the grain size and carbide distributions of the toroids produced by the two processing techniques, are discussed in terms of the various operations, temperatures, and strain rates associated with the two different processing schedules. The Press-Formed toroid had a higher yield strength than the HERF processed toroid which was attributed to the finer cell size of the PF processed toroid.

INTRODUCTION

High-Energy-Rate Forging (HERF) has been used extensively to fabricate complex shapes, with desirable mechanical properties, from stainless steel plate and bar stock. Recently, similar shapes with comparable properties have been produced by a Press Forming (PF) process. This study was performed to determine if structural differences existed in the toroids produced by the high-strain rate HERF and the low-strain rate PF processes and/or the thermomechanical cycles associated with HERF and PF processing, and to establish correlations between the microstructural observations, dislocation substructure, and the mechanical properties of the HERF and PF processed toroids. The material examined was the nitrogen strengthened, austenitic 21-6-9 stainless steel; also identified as Nitronic 40.

EXPERIMENTAL PROCEDURE

The compositions of the two heats of the vacuum-induction melted/electroslag remelted (VIM/ESR) 21-6-9 stainless steel investigated are given in Table 1. These alloy compositions fall within the WR procurement specifications included in Table 1.

The shapes examined were outer ring toroids either HERF or PF processed from 44.5-mm ($1\frac{3}{4}$ in.) thick plate of the compositions listed in Table 1. Metallographic sections for light microscopy and thin sections for transmission electron microscopy were removed from the toroids in the orientation shown in Figure 1. The metallographic sections were mechanically polished and etched electrolytically in a 5% ammonium persulfate solution at 4 volts for 15 seconds. The thin sections were ground to between 0.08 mm (0.003 in.) and 2.54-mm (0.10 in.), and electropolished in either a chromic acetic acid electrolyte at room temperature or a perchloric acid-methanol electrolyte at $-30\ ^\circ\text{C}$.

The HERF and PF processing differed significantly in the type and number of forming operations and the temperature at which the operations were performed. Tables 2 and 3 list the detailed forming schedules for the PF and the HERF processing, respectively. Also included in these tables are the nominal furnace temperatures at which the various operations were performed. For calculation of the ratio T/T_m (forming temperature, T , and melting temperature, T_m), a melting temperature characteristic of the solidus or lower temperature end of the melting range of WR specified 21-6-9 was taken as $2470\ ^\circ\text{F}$ ($1355\ ^\circ\text{C}$).¹ Apart from differences in type, number, and temperatures of operations between the HERF and the PF processing, there is a significant difference in strain rate associated with the two processes; that is, $800\ \text{cm}/\text{cms}^{-1}$ for HERF processing, and $0.5\ \text{cm}/\text{cms}^{-1}$ ⁽²⁾ for PF processing.

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TABLE 1. Compositions (%) of HERF and PF Processed Stainless Steel

Ident.	Processing	Cr	Mn	Ni	N	C	Al	Si	P	O (ppm)	S
Heat No. 7611 (MC-105260) 1-3/4 CRP	PF	19.3	9.09	6.77	0.30	0.029	0.001	0.34	0.017	25	<0.003
Heat No. 4568 (MC-104624) 1-3/4 CRP	HERF	19.5	9.50	6.75	0.29	0.024	NA	0.27	<0.02	28	0.004
WR Spec.	No. P-14110	19-21.5	8-10.0	5.5-7.0	0.2-0.3	0.02-0.04	0.017	0.3-0.7	0.20 Max.	70	0.015 Max.

FIGURE 1. Press Formed 21-6-9 Outer Toroid Showing Orientation of Samples Taken for Light Micrographs and TEM Thin Films

TABLE 2. Press Forming Processing Schedule

Operation	Temperature		T/Tm*
	°F	°C	
1. Hot cross roll plate, water quench	2000-1330	1095 to 720 °C	0.84-0.60
2. Solution treat -0.75 hr water quench	2000	1095	0.84
3. Cold cross roll	RT	-	-
4. Cut blanks	RT	-	-
5. Anneal and water quench	1500	815	0.67
6. Warm form - water quench	1000	540	0.50

*Assume Tm = 1355 °C (1628 °K) (see reference 1)

TABLE 3. HERF Processing Schedule

Operation	Temperature		
	°F	°C	T/Tm
1. Pancake formation - 5 blows. Water quench after each blow.	1850	1010	0.79
2. Cup formation - 2 blows. Water quench after each blow.	1850	1010	0.79
3. Machine bottom from cup.	RT	—	—
4. Height adjustment - 1 blow. Water quench	1850	1010	0.79
5. Curling over mandrel - 2 blows. Water quench after each blow.	1550	840	0.68
6. Anneal - 7 min. stress relieve	1550	840	0.68

Mechanical properties of the HERF processed 21-6-9 stainless steel examined in this report range between 730 to 780 MPa (107 to 113 ksi) for the 0.2% offset yield strength, 860 to 885 MPa (125 to 128 ksi) for the ultimate strength, and 41 to 46% for the total elongation. Typical mechanical properties of the PF processed 21-6-9 stainless steel examined in this report range between 756 to 927 MPa (110 to 135 ksi) for the 0.2% offset yield; 969 to 1051 MPa (143 to 153 ksi) for the ultimate strength; and 22 to 29% for total elongation. Results of mechanical tests used to establish these ranges are listed in Table 4.

RESULTS AND DISCUSSION

Microstructure

Figures 2 and 3 show the microstructures of the HERF and the PF processed 21-6-9 stainless steel, respectively. There are significant differences in the grain size and carbide distributions produced by the two different processing methods.

The grain size of the HERF material is ASTM No. 5½ and is significantly coarser than the PF structure which had mixed ASTM grain sizes of 7 and 9. The coarser grain size of the HERF material is probably a result of spontaneous recrystallization and grain growth accompanying the hot work operations at 1010 °C (1850 °F), whereas the finer grain size of

TABLE 4. Mechanical Properties of HERF and PF Processed 21-6-9 Stainless Steel

Condition	0.2% YS		Ultimate		Total Elongation (%)
	ksi	MPa	ksi	MPa	
Annealed Sheet	52	359	106	732	71
PF*	120	814	153	1051	22
	122	827	153	1051	25
	127	861	143	982	28
	135	927	151	1037	25
	117	804	143	996	26
	110	756	141	969	29
	116	797	146	1003	25
	121	831	153	1051	25
HERF**	124	852	147	1010	24
	107	737	127	873	41
	113	781	128	886	41
	107	272	125	860	46
	114	782	128	880	41

*Mechanical properties measured on specimens from parts processed identically to those examined in this report.

**From specimens described in this report.

the PF material is probably a result of recrystallization during the 815 °C (1500 °F) annealing following the room temperature cross rolling operation. The final deformation steps in both processes produced the intragranular dislocation substructure described in the following sections of this report, but did not substantially change the grain boundary configurations developed by recrystallization in the preceding operations.

The carbide distribution revealed in Figures 2b and 3b by etching is assumed to be associated with the formation of $M_{23}C_6$ chromium carbides that is well documented in the stainless steel literature.³ The carbides in the HERF processed steel are primarily concentrated on the austenite grain boundaries. In 21-6-9 stainless steel, $M_{23}C_6$ carbide formation at grain boundaries follows C-curve kinetics with a nose or minimum formation time at about 760 °C.^{4,5} Therefore, the carbides in the HERF processed steel have formed after the final recrystallization; probably on heating to 840 °C for the curling operation.

2(a). 100X

2(b). 500X

FIGURE 2. Microstructure of HERF Processed 21-6-9 Stainless Steel. Light micrographs, 5% ammonium persulfate electrolytic etch.

FIGURE 3. Microstructure of PF Processed 21-6-9 Stainless Steel. Light micrographs, 5% ammonium persulfate electrolytic etch.

3(a). 100X

3(b). 500X

The carbide particles in the PF processed steel are arrayed in what appears to be a coarse grain boundary network, but are, in fact, not associated with the final fine-grain boundary structure. The coarse carbide particle network decorates or marks the position of grain boundaries produced during the solution treatment at 1095 °C (2000 °F). It is unlikely that the carbides formed during the water quench from the solution treatment and, therefore, they must have precipitated on heating to the 815 °C (1500 °F) annealing temperature prior to the onset of recrystallization. The linear array of carbides found in Figure 3b is caused by the carbides forming upon heating through 760 °C temperature range to the 815 °C annealing temperature and precipitating on deformation bonds which were produced during cold cross rolling.

Substructure: General Observations

Two types of dislocation substructure typical of high stacking fault energy alloys were observed in the thin foils removed from the HERF and PF processed shapes. The one type of substructure is described in the literature as being composed of cells and was observed in both the HERF and the PF processed specimens. The other type of substructure consists of subgrains and was observed only in HERF processed specimens. The dislocation cells are characterized by dislocation-free interiors and relatively thick walls that are made up of a high density of tangled dislocations. Dislocation subgrains also are characterized by almost dislocation free interiors, but tend to be larger than the cells and have well defined boundaries accommodating lattice misorientations between adjacent regions within the grains. Cells tend to form during cold or warm working between room temperature and 0.5 Tm or 0.6 Tm, whereas subgrains tend to form at higher temperatures.^{6,7}

The results of the measurement of dislocation cell sizes in both HERF and PF processed specimens are presented in Table 5. Cell size was taken as the largest dimension of the dislocation free area of a cell and, therefore, would be somewhat smaller than if measured by an intercept technique where the width of the cell wall would also be taken into account. Other investigators⁶ also multiply the

TABLE 5. Cell Sizes in HERF and PF Processed Specimens

Processing	Plate	Cell Size (μm)	Number of Cells Measured
HERF	717	0.3	8
HERF	718	0.23	4
HERF	720	0.27	2
HERF	734	0.24	5
HERF	745	0.31	4
HERF	747	0.23	3
PF	341	0.16	8
PF	350	0.15	4
PF	616	0.19	3
PF	674	0.20	3
PF	684	0.12	1

Note: Cell size measured was the maximum dimension of the dislocation free area.

results of intercept measurements by the factor 1.68 (assuming that the cells are space-filling Kelvin polyhedra). The measurements show that the cell size of the PF processed specimens is definitely smaller than the HERF processed specimens, despite the small number of measurements. The cell sizes measured are also consistent with those measured after tensile deformation of 316 stainless steel. Michel, et al.,⁶ found that the cell size increased continuously from 0.32 μm to 0.67 μm as the deformation temperature increased from 21 °C to 593 °C, the temperature range over which cell formation occurred. Above that temperature subgrains significantly larger in size developed in the 316 stainless steel. Our average cell sizes, multiplied by the 1.68 factor to permit comparison with the results of Michel, et al.,⁶ are 0.49 μm and 0.27 μm for HERF and PF specimens, respectively. As noted earlier, the HERF specimens also developed a subgrain structure in addition to the cell substructure. The subgrains were irregular in shape and are estimated to be a factor of 2 to 3 times coarser in their smallest dimension than the cells. Examples of the various dislocation substructures are presented in the following sections.

FIGURE 4. Grains and Substructure in HERF Processed 21-6-9 Stainless Steel

Substructures Developed by HERF Processing

Figure 4 is a transmission electron microscope (TEM) micrograph of HERF processed stainless steel taken at relatively low magnification. The large angle boundary configurations shown in the light micrographs of Figure 2 are also visible in Figure 4 and, in addition, a dense dislocation substructure is visible throughout the austenite grains.

Figures 5, 6, and 7 show examples of well-developed subgrains within the grains of the HERF processed 21-6-9 stainless steel. The subgrain boundaries are

narrow and in good contrast because of the concentration of dislocations required to accommodate the lattice misorientation between adjacent grains. Figure 5 shows an area of subgrains containing only incipient interior dislocation cells, whereas Figure 6 shows well-developed cells and tangles together with subgrain boundaries. Figure 7 shows the same area of a thin foil in two different orientations to the incident electron beam. The different orientations clearly show that an orientation difference exists between the central subgrain and its surroundings. Again, tangled dislocations have formed a cell structure within the subgrains.

FIGURE 5. Subgrains and Subgrain Boundaries in HERF Processed 21-6-9 Stainless Steel.

Details of the dislocation cell structure formed in the HERF processed shape are shown in Figures 8 and 9. Figure 8 shows bright and dark field micrographs taken adjacent to a large angle grain boundary. There are areas that are almost free of dislocations, but much of the structure consists of loosely tangled dislocations that form the boundaries of the cells. The dislocations are jogged and some dislocation loops are present. This is evidence⁸ for the large amount of dislocation intersection that, together with cross slip, contributed to the formation of the dislocation cell structure. Figure 9 also shows jogged dislocations and other dislocation debris that are a result of dislocation intersection.

Many dark particles appear to be present at relatively low magnification, as used to show the substructures in Figures 5 through 7. Higher magnification micrographs shown in Figures 8 and 9 resolve many of these dark particles as the dislocation loops or debris referred to above, and establish that the matrix is essentially free of any precipitate particles.

Substructures Developed by PF Processing

Figures 10 through 13 show the substructure of the PF processed 21-6-9 stainless steel. Only dislocation cells were noted, and in some areas, as

FIGURE 6. Examples of Areas With Mixed Cell and Subgrain Structures in HERF Processed 21-6-9

FIGURE 7. Subgrain in Foil Tilted in 2 Different Orientations With Respect to Incident Beam. HERF specimen.

FIGURE 8, Details of Dislocation Structure in HERF Processed Specimen, Bright Field.

shown in Figure 10, the cells appeared to be defined by high densities of dislocations on well-defined planes. Details of the dislocation structures are shown in Figures 11 and 12. Extensive jogging of the dislocations and dislocation loops, formed as

the result of dislocation intersection are apparent. The dislocation arrays appear to be more regular in Figure 12 than those in Figure 11, perhaps as a result of a greater degree of recovery in the area shown in Figure 12.

FIGURE 8 (continued). Details of Dislocation Structure in HERF Processed Specimen, Dark Field.

Figure 13 is a dark field exposure included to show an area where dissociation of the perfect dislocations has produced extended dislocations made up of partial dislocations separated by stacking faults. The faults may be identified by the parallel bright fringes

visible in Figure 13. The dissociation of dislocations, as noted, is a characteristic of low stacking fault energy alloys, and is associated with planar dislocation arrays because of the reduced ability of extended dislocations to cross-slip.⁹ Our observations show

FIGURE 9. Details of Dislocation Structures in HERF Processed Specimen

that the deformation behavior of the 21-6-9 processed both by HERF and PF is predominately that produced by undissociated dislocations that are capable of easy cross slip and intersection, and hence produce tangles and cells as opposed to planar arrays. Nevertheless, the observation of some dissociated dislocations indicates that the 21-6-9 examined here may have a stacking fault energy just above that required for extensive dissociation.

Odegard, et al.,¹⁰ have shown a transition from tangled dislocation arrays to planar arrays of extended dislocations as the nitrogen content of 21-6-9 was increased from 0.12 to 0.47 percent. Further, they¹⁰ showed that the reduction of area (RA) of 21-6-9 when exposed to hydrogen decreased significantly when planar dislocation arrays were present, with the largest discontinuity developing between 0.24 and 0.31 percent nitrogen. The latter

FIGURES 10(a) and (b). Cell Structure in PF Processed 21-6-9 Stainless Steel

FIGURE 11. Details of Dislocation Cell Structure in PF Processed 21-6-9 Stainless Steel

comments, together with our observations, indicate that the 21-6-9 heats under investigation here, containing 0.29 and 0.30 percent nitrogen, although consisting primarily of substructures formed by undissociated dislocations, are on the borderline of dislocation dissociation characteristic of low stacking fault energy stainless steel. Generally, small subgrain sizes such as observed in this investigation, are also considered to be associated with low stacking fault energies.⁷

DISCUSSION

The dislocation substructures described in the previous sections can be qualitatively related to the last deformation stages in the HERF and PF processing schedules. Two important differences exist between the two types of processing: differences in the temperatures of the last steps, 0.50 Tm versus 0.68 Tm for the PF and HERF processes steel respectively, and differences in

FIGURE 12. Dislocation Substructure in PF Processed 21-6-9 Stainless Steel

strain rate, 5×10^{-1} cm/cm/sec versus 8×10^2 cm/cm/sec for the PF and HERF processing respectively.

Generally, cold working of metals is defined as deformation below $0.4 T_m$ and hot working as deformation above $0.5 T_m$.⁷ However, these limits are affected by strain rate with increasing strain rates displacing the temperature range of cold work to

higher temperatures. Also the transition from cold to hot work may extend over an appreciable range, up to $0.5 T_m$ or $0.6 T_m$, a transition range which is sometimes referred to as warm working. The operative dislocation mechanisms during cold and hot working are relatively insensitive to strain rate provided the strain rate is kept below 10^3 S^{-1} .⁽⁷⁾

Both HERF and PF processed specimens contained typical cold worked substructures consisting of cells

FIGURE 13. Partial Dislocations and Stacking Faults on PF Processed 21-6-9 Stainless Steel. Dark field micrograph.

with walls made up of tangled dislocations. This result is reasonable for the PF processed specimens where the temperature of the final deformation, $0.50 T_m$, is well within the accepted range for cold or warm working. However, the observation of a cell structure on the HERF processed specimens is surprising because of the high temperature, $0.68 T_m$, of the final deformation operation. Apparently the very high strain rate of the HERF processing is sufficient to prevent the diffusion-controlled climb processes necessary for the development of a hot work substructure during the very last HERF deformation. The cells in the HERF processed specimens were somewhat larger than those noted in the PF

processed specimens, a difference that may be accounted for by differing amounts of cold work and/or the deformation temperature.^{6,7}

The observation of the well-developed subgrains and subgrain boundaries in the HERF specimens shows that dynamic recovery typical of hot work occurs at some stage of the final processing prior to the introduction of the cell structure. There are a number of explanations for the mixed subgrain-cell substructures observed. One possibility is that, after the formation of the subgrains at a higher temperature, the final blow of the curling operation may have been performed at a temperature below the nominal

furnace temperature of 840 °C, perhaps closer to 0.5 T_m where cell structures readily form by cold working dislocation mechanisms.^{6,7}

Assuming that the nominal temperatures of the operation are close to the actual temperatures of deformation, it is possible that the high strain rate of the HERF process overrides the effect of temperature and does, in fact, produce a cold work type of substructure at the HERF curling temperature of 0.68 T_m. Both blows of the curling operation could, therefore, have introduced the cell substructure, while the subgrain may have been introduced during the height adjustment operation at 1010 °C or 0.78 T_m. Still another possibility is that the first blow of the curling operation introduced the subgrain structure and the second blow introduced the cell structure.

Without additional information, or experimentation, the exact stages of the development of the dual-substructure are difficult to state. Additional work should include a closer monitoring of the actual deformation temperatures, and the amount of deformation at each stage; better understanding of the interrelationships between amount of deformation, high strain rate, and temperature on substructure development; and an estimation of the role that factors such as adiabatic heating, important to HERF processing, would play in substructure development. Examination of the substructure after each processing step would directly resolve the origin of the various types of substructure in the HERF processed specimens.

The high strength of the 21-6-9 stainless steels as listed in Table 4 can be directly related to the presence of the previously described substructures. Where dislocation cells are present, the flow stress and cell size have been related to the high dislocation density in the cell walls. A general equation¹¹ relating flow stress and cell or subgrain size is:

$$\tau = \tau_0 + K G b d^{-m}$$

where τ_0 is the lattice friction stress, K is a constant, G is the shear modulus, b is the Burger's vector, and m is a constant that depends on the nature of the substructure boundary. Subgrain boundaries with their greater perfection and misorientation are considered to behave like large angle boundaries, and the value of m associated with their effect on

strength is $\frac{1}{2}$; as in the Hall-Petch equation for large angle boundaries. Frequently, values of $m = 1$ ⁽¹¹⁾ have been obtained for cell structures. In cases where both cells and subgrains exist, $m = 1$ has been measured when the boundaries are submicron in size, with a shift to $m = \frac{1}{2}$ when most of the cell walls are replaced by more widely spaced subgrain boundaries.

The submicron size of the cell substructure measured for both the HERF and PF processed 21-6-9 stainless steel indicates that the flow strength less the friction stress is inversely proportional to the cell size. The finer cell size of the PF processed shape should, therefore, produce a higher flow stress than that of the HERF processed material; a conclusion that is supported by the significantly higher yield strengths measured for the PF processed stainless steel specimens.

CONCLUSIONS

The following conclusions, based on the work performed, apply to HERF and PF processed 21-6-9 stainless steel formed according to the schedules listed in Tables 2 and 3.

1. Recrystallization after room temperature rolling produces final mixed grain sizes of ASTM No. 9 and 7 in PF processed toroids. Carbides are formed on austenitic grain boundaries prior to recrystallization, but are not associated with the final grain boundary structure.
2. The final grain size of ASTM No. 5½ was produced in HERF processed 21-6-9 by recrystallization and grain growth during intermediate processing stages performed at 1010 °C. Carbides are present on the final grain boundary structure.
3. The dislocation substructure of PF processed specimens consists of cells with boundaries made up of loosely tangled dislocations. This substructure is produced by dislocation cross slip and dislocation intersection characteristic of cold work, and is consistent with a final deformation temperature of 0.50 T_m.
4. The substructure of the HERF processed specimens consists of subgrains formed by dynamic recovery typical of hot working

deformation, and of dislocation cells characteristic of cold work. The mixed substructure is a result of the interplay between the high strain rate of HERF deformation and the high temperatures, 0.68 to 0.78 T_m, at which the final deformation is performed.

5. The cell size of the PF processed specimens (0.16 μm), is smaller than the cell size of the HERF processed specimens (0.29 μm).
6. The higher yield strength of PF processed 21-6-9 correlates well with its finer cell structure in accord with literature reports that the strength of metals with submicron size cell structures is inversely proportional to cell size.
7. Most dislocations observed were undissociated, but some dissociation into partials and stacking faults was noted. This observation, together with the very fine cell size, indicates that the stacking fault energy of the 21-6-9 examined was on the border of that associated with extensive dislocation dissociation and planar dislocation arrays.

FUTURE WORK

Applications for steel forgings such as described in this study require that work be retained in the complex shapes to increase the strength of the alloy. The nature of the deformation and the metallurgical substructure equivalent to the retained work plays an important role in establishing the properties of the forgings. Adequate performance of the alloy requires that the structure remain metallurgically stable against stresses and environmental conditions seen in long term service. It is possible that proper deformation processing could produce a dislocation substructure which would enhance environmental compatibility. Many other questions remain; such as, what controls are necessary in deformation processing to prevent undesirable precipitation and dislocation substructure?

More information is needed on the relationship between deformation processing and substructures developed in stainless steels such as 21-6-9. Complete characterization of structure requires light metallography and electron metallographic techniques capable of resolving the elements of structures, such as the dislocation arrays described in this report.

Initial results show that substantial differences exist between HERF and PF processing of identical shapes. This study will continue in order to learn what these differences mean in terms of development of desirable dislocation substructures, optimum deformation processing of 21-6-9, and the effect of process controls on mechanical properties.

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