EVOLUTION OF MICROSTRUCTURE DURING HOT ROLLING

OF INCONEL® ALLOYS 625 AND 718

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Abstract

With today's requirements for increased productivity and tighter mechanical property and microstructural specifications imposed by customers, a good understanding of microstructural development and control is required. At Inco Alloys International Inc. (IAII), a pilot mill and a production mill were used concurrently to process INCONEL alloys 625 and 718. Finite element software was used for increased understanding of thermomechanical processing (TMP) during flat rolling. As-rolled grain size and volume fraction of recrystallization were analyzed. It was determined that finishing temperature plays an important part in recrystallized grain size and that volume fraction of recrystallized grains is dependant on many factors, especially strain rate and temperature.

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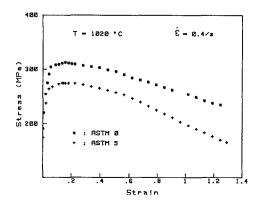
Introduction

Many papers have been written about processing materials isothermally.¹⁻⁸ Recrystallization maps have been published and TMP ranges have been recommended. These recommendations may be very effective for single blow forging or other isothermal or nearly isothermal processes. Production rolling is not isothermal. Machine capacities, such as load and roll speed, limit the process as well. Some variables for consideration are material cooling during processing, reheat times (if necessary), maximum strain rates available and influence of these factors on mill loads.

Control of Microstructure

It is well known that microstructural control is of paramount importance concerning mechanical properties of materials. Microstructure also plays an important role in processing materials at elevated temperatures. Fine-grained materials have greater hot ductility and require lower mill processing loads, when dynamic recrystallization occurs, than materials with larger grains. Figure 1 shows the effect of grain size on flow stress during hot deformation of alloy 718.

Hot deformed materials can be separated into 3 groups with respect to recrystallization (RXN); fully recrystallized (RXD), partially recrystallized (PRXD), and unrecrystallized (UNRXD). (See Figure 2). In general, it is necessary to avoid partial recrystallization (PRXN) to meet customer specifications. If some UNRXD grains exist from a previous deformation, they may not recrystallize with further deformation. Subsequent deformations may only refine the already RXD areas leaving large UNRXD grains surrounded by a fine-grained necklace structure, as illustrated in Figure 3. Fine, uniform grain structure can be achieved by deforming materials below the RXN start temperature (controlled rolling) followed by a RXN heat treatment just above the static RXN temperature. This process is similar to cold rolling plus annealing. Achieving fine, uniform structures in the as-hot-rolled condition, requires large deformations at temperatures just above the RXN temperature.



Partial RXN No RXN Strain

Figure 1 Influence of initial grain size on flow stress.¹

Figure 2 Schematic of recrystallization behavior of alloys. 10

In single deformation (ϵ =0.8), hot compression tests⁸, alloy 625 exhibits a temperature threshold below which partial recrystallization is inevitable independent of the strain rate, Figure 4.⁸ There is also a strong dependence of RXD grain size (RXD GS) on deformation temperature.

For alloy 718, as-rolled, RXD GS is highly dependent upon deformation temperature, strain and strain rate. G. Camus¹ showed that higher strain rates produce larger RXD grains with greater volume fraction of RXN. A similar relationship between RXD GS, finishing temperature and strain was seen for alloy 625. ¹² (Strain rate effects were not investigated.) Figure 5 illustrates the effect of strain rate and deformation temperature for alloy 718.

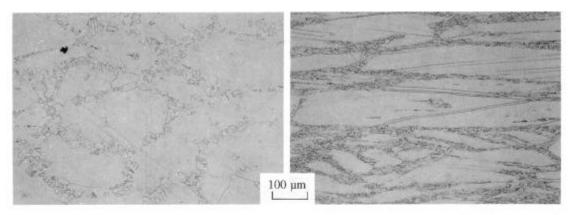


Figure 3 Micrographs of INCONEL alloy 625 flat rolled 25% in 2 passes (a) and 63% in 5 passes (b).

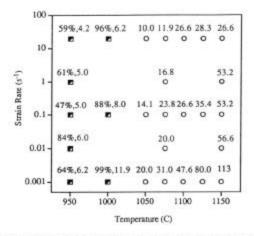


Figure 4 Microstructural map for as-deformed, alloy 625 deformed to a true strain of 0.8; percentage of RXN is shown for PRXD microstructures and RXD GS is shown in µm.8

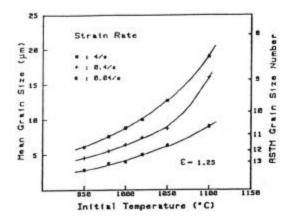


Figure 5 Variation of dynamically RXD GS as a function of the initial testing temperature and strain rate for alloy 718.

It has been shown that volume fraction of RXD grains, in alloy 718, is dependent upon initial grain size, temperature, strain and strain rate. Higher fraction RXN occurs at higher temperatures, with finer initial grain size, with greater amounts of deformation and at higher strain rates.

Current Work

The current work focuses on the microstructures produced on a pilot rolling mill, located in the IAII Technology Processing Center (TPC), and a production rolling mill, located in the IAII Bar and Wire (B&W) department. Major differences in process capability between the two mills are maximum roll speed and grooved rolls for edge rolling. Features of these mills are given in Table I.

Table I Features of production and pilot mills.

Feature	B&W 23" Breakdown Mill	TPC 20" Pilot Mill	
Manufacturer	Mesta	Birdsboro	
Roll Diameter	580 mm (23")	500 mm (20")	
Maximum Separating Force	4500 kN (1.0 M lbs.)	8100 kN (1.8 M lbs.)	
Power	1500 kW (2000 hp.)	280 kW (375 hp.)	
Max roll speed	120 rpm.	15 rpm.	
Roll Design	edge passes & bull head	flat	

Experimental Procedure

Alloy 625 Recrystallization Map

The pilot mill was used to produce a RXN map by flat rolling 35mm (1.4") thick plates in 12.5% (of the original thickness) increments at starting temperatures from 927°C to 1204°C (1700°F to 2200°F). Samples were soaked at temperature then rolled to final thickness in 1 to 6 passes.

Production Mill Rolling

Production billets, 150x200mm (6x8") cross section, of alloys 625 and 718 were rolled to 70x90mm (2.75x3.625") cross sections via standard mill procedures. This procedure included heating to $1177^{\circ}C$ (2150°F) for alloy 625 and $1121^{\circ}C$ (2050°F) for alloy 718 and rolling according to the computer controlled rolling schedule. The last 6 passes are described in Table II. The grooves listed are bull head (BH) or flat rolling area of the rolls and edge pass grooves of nominal width x (of xE in Table II), in inches.

Pilot Mill Rolling

Material was acquired from the end of each cog as rolling stock for the pilot mill sequences. Samples, 76x76x150mm (3x3x6"), were cut from these cog ends then heated to the above rolling temperatures and rolled using flat rolling and a flip-up rolling technique. This technique is performed by rolling on a flat mill and rotating the piece along the longitudinal axis between passes. This helps to maintain a near-square cross-section rather than an extremely rectangular cross section as obtained in flat rolling. These rolling sequence schedules are also given in Table II. The six passes shown approximate the reduction in areas of the last 6 passes on the B&W mill to allow microstructural comparison to the production material.

Computer simulated rolling

To characterize the time-temperature-deformation history of the flat rolling sequence of the recrystallization map, rolled from 1204°C (2200°F), finite element analysis (FEA) was performed to simulate the six pass deformation. The simulation used a coupled thermomechanical analysis with thermal interactions between the roll and sample and interpass heat transfer considered. The commercial FEA software DEFORM*.13 was used to perform the simulations.

^{*} DEFORM is a trademark of Scientific Forming Technologies Corporation.

<u>FEA Formulation and Mesh</u> DEFORM utilizes a flow formulation common in finite element methods for simulating metal flow. The method solves for nodal velocities and models rigid, viscoplastic material behavior. Elastic effects are assumed to be negligible. Four-node quadrilaterial elements were used.

The geometry of flat rolling allows for a plane-strain assumption which is modelled in 2D. The size of the problem is further reduced by assuming planar symmetry at the center of the roll sample. Approximately 500 elements were used to discretize the sample.

<u>Material Properties and Boundary Conditions</u> Material properties for alloy 625 were obtained from several sources. Elevated temperature flow data, generated by Concurrent Technologies Corporation⁶, was used to characterize material flow; thermal properties were obtained from IAII literature.¹⁴

The heat transfer between the roll and workpiece was characterized by a heat transfer coefficient of 108 kW/m²/K and a constant roll temperature of 200°C. These values were based on a review of other FEA roll models. A convection coefficient of 0.0227 kW/m²/K is used to model heat transfer at surfaces not in contact with the roll. The value is a DEFORM default.

Metallography

Metallographic work was performed on samples of rolled bar. Grain size measurements were performed utilizing the microstructural comparison method in ASTM E112 on samples, polished then etched with bromine. Amounts of RXN were estimated.

Table II Bar and Wire production mill and Technology Processing Center pilot mill rolling schedules.

B&W Mill 150x200mm (6x8") starting size		% Red.	TPC Mill 76x76mm (3x3") starting size		
Pass No.	Groove		Pass No.	Roll Gap*, mm (in.)	
				Flat Rolling	Flip-up Rolling
6	ВН	12.1	1	67 (2.64)	67 (2.64)
7	3.5E	4.2	2	64 (2.53)	73 (2.87)
8	ВН	13.8	3	60 (2.35) 55 (2.18)	62 (2.46) 58 (2.27)
9	3E	21.7	4	49 (1.94) 43 (1.71)	65 (2.56) 57 (2.25)
10	ВН	16.0	5	36 (1.43)	53 (2.09) 49 (1.91)
11	2.5E	19.3	6	29 (1.16)	52 (2.03) 46 (1.82)

^{* --} TPC Mill passes containing 2 roll gaps used 2 passes for a single B&W Mill pass, due to mill constraints.

Alloy 625 RXN Map

According to the FEA model, run for alloy 625 heated to 1204°C (2200°F), the roll-contact-chill effect caused a 550°C (990°F) temperature gradient within 2.5mm (0.10") of the surface in the first pass. This gradient was reduced to approximately 110°C (200°F) during the 4 second delay prior to the second pass. Similar roll contact gradients were seen for the other passes though the gradients after the 4 second interpass time reduced each pass until the gradient was less than 10°C (18°F). This decrease in gradient can be attributed to the reduced thickness of the plates. Strain gradients were observed with a peak strain located between 25 and 30% of the thickness from the surface of the plate. The strain gradient increased with each pass while the location of the peak strain remained relatively stationary. This peak strain can be attributed to roll geometry, friction and temperature gradient. There was no microstructural effect associated with this region of higher strain. Figure 6 shows the temperature and strain gradients immediately after the second roll pass, as predicted by DEFORM.

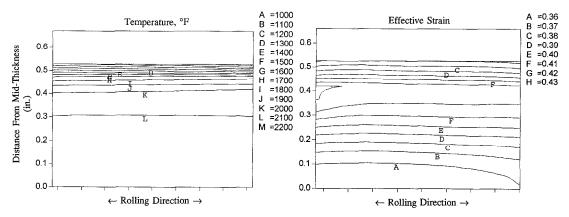


Figure 6 DEFORM predicted temperature and strain profiles immediately following the second pilot mill roll pass.

For the above reasons, the microstructures analyzed in this portion of the current work are from the center of the thickness on the longitudinal surface. Etched samples of the RXN map study showed a large variation in microstructure across the thickness of the specimen, with largely UNRXD surface zones and more RXD centers. The RXN map for alloy 625 under non-isothermal, flat rolling conditions is given in Figure 7.

Alloys 625 and 718 Rolling

<u>Finishing Temperatures</u> The surface finishing temperatures of the materials were measured using an optical pyrometer. Alloy 625 finishing temperatures were 927°C (1700°F) for pilot mill flat rolling and 871°C (1600°F) for flip-up rolling. Alloy 718 finished off the B&W mill at 999°C (1830°F) and off the pilot mill at 904°C (1660°F) for flat rolling and 871°C (1600°F) for flip-up rolling.

Microstructural Evaluations Grain size measurements were made on the starting billets in the annealed condition for alloy 718. As-rolled grain sizes are reported for alloy 625 in Table III and for alloy 718 in Table IV. Data is given for the starting annealed grain size as well as surface and center areas of the production mill rollings and pilot mills trials. Micrographs of as-rolled microstructures for B&W rolling and pilot, flat rolling are given in Figures 8 and 9 for alloy 625 and Figures 10 and 11 for alloy 718.

ture	1204 (2200)	45% 5 1202 ^{FEA}	80% 6 1190	80% 9 1145	80% 11 1087	75% 12 1021	80% 12.5 956
Temperature	1149 (2100)	5% 8.5	40% 8	5% 11	8% 12.5	50% *	30% *
Rolling To	1093 (2000)	<5% 12	<5% 10	<5% 12	X	<5% *	Х
ng Rol	1038 (1900)	<5% 11.5	Х	<5% *	<5% *	Х	х
Starting	982 (1800)	Х	X	X	X	Х	X
		12.5	25.0	37.5	50.0	62.5	75.0

Total Deformation (12.5% / roll pass)

FEA -- FEA predicted rolling temperatures for 1204°C heated samples.

* -- Previously RXD grains are twinned (approximately ASTM # 12.5)

X -- indicates no RXN

Figure 7 Non-isothermal RXN map for flat rolled alloy 625.

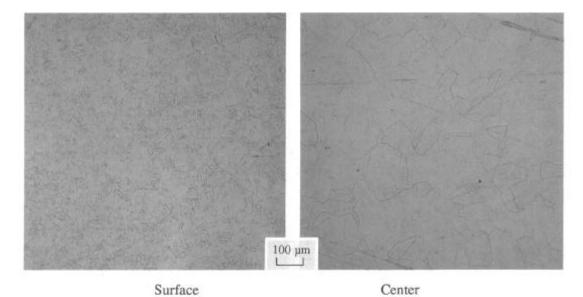


Figure 8 Optical micrographs of B&W mill rolled alloy 625 at surface and center locations.

Discussion

Nowhere on the alloy 625 RXN map, Figure 7, is complete RXN indicated. This is attributed to the significant cooling, from roll contact, of the material as further strain is applied and to the relatively low roll speed of the TPC mill, a strain rate effect as described earlier. Microstructures of the TPC rollings from 76x76mm (3x3") cross section, Figures 8 and 9, show near full RXN at mid-thickness which indicates that there is a sample size effect between these two rolling sequences. The alloy 625 RXN map also shows a line below which RXN does not occur. This barrier seems to be associated with the RXD GS of ASTM # 12.5 and a rolling temperature of slightly below 1038°C (1900°F).

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Table III Microstructural evaluation of the starting and as-rolled samples of INCONEL alloy 625.

INCONEL alloy	Starting mat'1 - annealed @ 1177°C (2150°F)/2hrs.		As Rolled				
625			Surf	ace	Center		
	MS	ASTM GS	MS	ASTM GS	MS	ASTM	
Production Mill Rolling	10 M TO		100% RXD	8	100% RXD	2.5	
Pilot Mill Flat Rolling	Uniform	0.5	5% RXD	12	<5% UNRXD	RXD 11	
Pilot Mill Flip-up Rolling	Uniform	0.5	35% RXD	10.5	100% RXD	11.	

Table IV Microstructural evaluation of the starting and as-rolled samples of INCONEL alloy 718.

INCONEL alloy	Starting mat'1 - annealed @ 1177°C (2050°F)/2hrs.		As Rolled				
718			Surf	ace	Center		
	MS	ASTM GS	MS	ASTM GS	MS	AST	
Production Mill Rolling	Uniform	0.5	100% RXD	9	100% RXD	4	
Pilot Mill Flat Rolling	Uniform	0	50% RXD	RXD GS 12	100% RXD	1	
Pilot Mill Flip-up Rolling	Uniform	0	20% RXD	RXD GS 11.5	100% RXD	1	

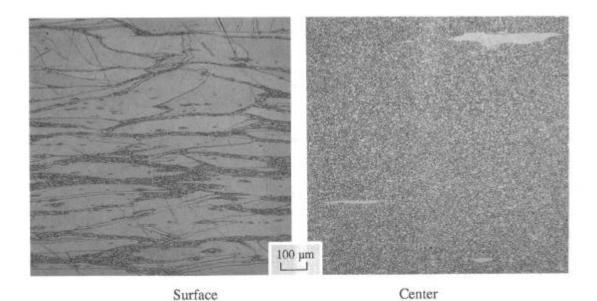


Figure 9 Optical micrographs of pilot mill rolled alloy 625 at surface and center locations.

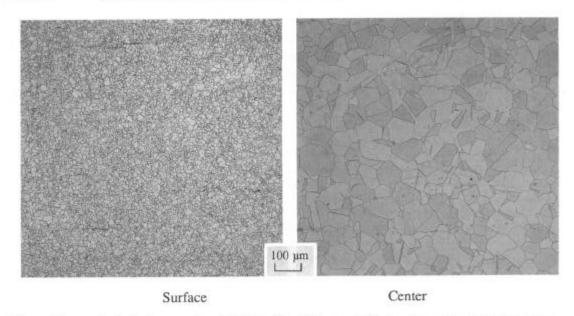
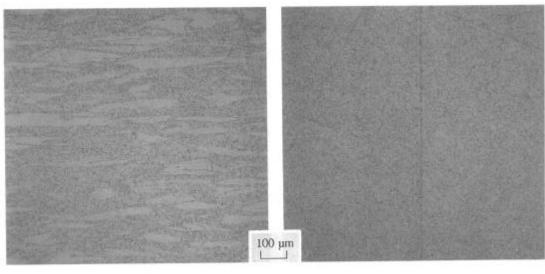


Figure 10 Optical micrographs of B&W mill rolled alloy 718 at surface and center locations.

The variation in amount of RXN across the 1149°C rolling temperature attests to the fact that amount of RXN is strongly dependent on many factors and is not readily predicted. The deformation sequence at 1204°C shows that once PRXN occurs it is difficult to recrystallize the UNRXD material even above the RXN temperature. Under further deformation, the temperature decreased from roll contact and the only RXN observed was further refinement of the already RXD areas. Possible contributors to this phenomenon include grain orientation, that most of the strain from subsequent passes ocurrs in the softer RXD grains and that the finer RXD grains recrystallize more readily than the larger UNRXD grains, a grain size effect.



Surface Center

Figure 11 Optical micrographs of pilot mill rolled alloy 718 at surface and center locations.

A cross-section / sample size effect has already been discussed with respect to the pilot mill rolling of alloy 625. When comparing the production mill rollings to those performed on the pilot mill there is inevitably a strain rate effect as well. The strain rates on the pilot mill are nearly an order of magnitude lower than those of the production mill. The combination of these two effects are clearly seen when comparing amount of RXN of alloy 625 and 718 rolled on the two mills, Tables III and IV.

Temperature Effects on RXD Grain Size

Alloy 625 In analyzing the alloy 625 hot compression data from Zhao, a correlation between RXD GS and deformation temperature is apparent. Figure 12 shows this data plotted in conjunction with RXD GS data from the current work. It is evident from Figure 12 that there are significant differences between the data plotted. These differences may be attributed to the effects of rolling rather than uniaxial compression, differences in chemistry, strain rate effects and the multi-pass history of the materials. Nevertheless, a strong correlation exists.

Alloy 718 Mataya³ performed four stroke compression tests, on alloy 718, at a constant true strain rate of 1.0 s¹. Figure 13 shows RXD GS data from Mataya plotted with respect to finishing temperature. Also plotted are alloy 718 grain-size data from the B&W rollings and pilot mill trials. Clearly, RXD GS is strongly dependent on finishing temperature. The regression line associated with data from this work is shifted due to higher strains than in Mataya's work. The difference in slope of this line is most likely due to grain coarsening during cooling which would be more prevalent at higher temperatures.

Conclusions

- Recrystallized grain size of INCONEL alloys 625 and 718 is highly dependent upon finishing temperature for typical rolling operations.
- There is a large sample size affect on the recrystallization behavior of non-isothermally hot rolled alloy 625.
- Roll speed can play a large role in the recrystallization behavior of hot rolled alloys 625 and 718.
- Prediction of amount of recrystallization will continue to be extremely difficult in multi-pass non-isothermal rolling of superalloy 625 and 718.

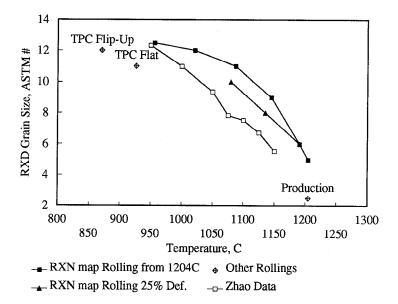


Figure 12 Recrystallized grain size as a function of deformation temperature for alloy 625. Data from Zhao⁸ and the current work.

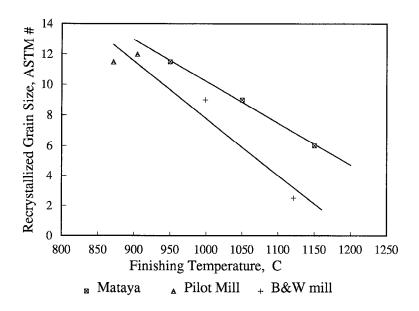


Figure 13 Recrystallized grain size as a function of finishing temperature for alloy 718. Data from Mataya³ and the current work.

References

- 1. G. Camus, B. Pieraggi, F. Chevet, "Hot Deformation and Recrystallization in Inconel 718," <u>Formability and Metallurgical Structure</u>, ed. A.K. Sachdev and J.D. Embury (Warrendale, PA: The Metallurgical Society, 1987), 305-325.
- 2. M.J. Weis et al., "The Hot Deformation Behavior of an As-Cast Alloy 718 Ingot," <u>Superalloy 718</u>, ed. E.A. Loria (Warrendale, PA: The Minerals, Metals & Materials Society, 1989), 135-154
- 3. M.C. Mataya, D.K. Matlock, "Effects of Multiple Reductions on Grain Refinement During Hot Working of Alloy 718," <u>Superalloy 718</u>, ed. E.A. Loria (Warrendale, PA: The Minerals, Metals & Materials Society, 1989), 155-178.
- 4. P.J. DiConza, R.R. Biederman, R.P. Singh, "Homogenization and Thermomechanical Processing of Cast Alloy 718," <u>Superalloys 718, 625, and Various Derivatives</u>, ed. E.A. Loria (Warrendale, PA: The Minerals, Metals & Materials Society, 1991), 161-171.
- 5. C.I.Garcia et al., "Microstructural Refinement of As-Cast alloy 718 Via Thermomechanical Processing," <u>Superalloys 718, 625, and Various Derivatives</u>, ed. E.A. Loria (Warrendale, PA: The Minerals, Metals & Materials Society, 1991), 925-941.
- 6. Concurrent Technologies Corporation, <u>Atlas of Formability Nickelvac 625</u>, National Center for Excellence in Metalworking, Johnstown, PA, 1993.
- 7. D. Zhao, P.K. Chaudhury, "Effect of Starting Grain Size on As-Deformed Microstructure in High Temperature Deformation of Alloy 718," <u>Superalloys 718, 625, 706 and Various Derivatives</u>, ed. E.A. Loria (Warrendale, PA: The Minerals, Metals & Materials Society, 1994), 303-313.
- 8. D. Zhao et al. "Flow Behavior of Three 625-Type Alloys During High Temperature Deformation," <u>Superalloys 718, 625, 706 and Various Derivatives</u>, ed. E.A. Loria (Warrendale, PA: The Minerals, Metals & Materials Society, 1994), 315-329.
- 9. George E. Dieter, Mechanical Metallurgy (McGraw-Hill, 1976), 195.
- 10. A.J. DeArdo, "Modern Thermomechanical Processing of Microalloyed Steel: A Physical Metallurgy Perspective," Microalloying '95, (Warrendale, PA: Iron and Steel Society, 1995), 15-33.
- 11. George E. Dieter, Mechanical Metallurgy (McGraw-Hill, 1976), 551.
- 12. L. Ferrer, B.Pieraggi, J.F. Uginet, "Microstructural Evolution During Thermomechanical Processing of Alloy 625," <u>Superalloys 718, 625 and Various Derivatives</u>, ed. E.A. Loria (Warrendale, PA: The Minerals, Metals & Materials Society, 1991), 217-228.
- 13. Scientific Forming Technologies Corporation, DEFORM-PC Primer, Columbus, OH, 1996.
- 14. INCONEL alloy 625 Technical Bulletin, (Huntington, WV: Inco Alloys International, Inc., 1985)
- 15. C. M. Sellars, "Computer Modelling of Hot-Working Processes," <u>Materials Science and Technology</u>, 1 (1985), 325-332.
- 16. W. C. Chen, I. V. Samarasekera, and E. B. Hawbolt, "Fundamental Phenomena Governing Heat Transfer During Rolling," <u>Metall. Trans. A</u>, 24A (1993), 1307-1320.