Effects of Cold Work and Annealing on Microstructure and Mechanical Properties of HAYNES® N-86 Alloy

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HAYNES® N-86 is a nickel-based high-performance alloy utilized for high temperature applications. In this study, HAYNES® N-86 was cold-rolled and annealed to examine the effects of those processes on the mechanical properties and microstructure. This allows for a better understanding of the relationship between processing and properties for this alloy, with the goal being to adjust the processing parameters to meet the mechanical property specifications more consistently.

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HAYNES International

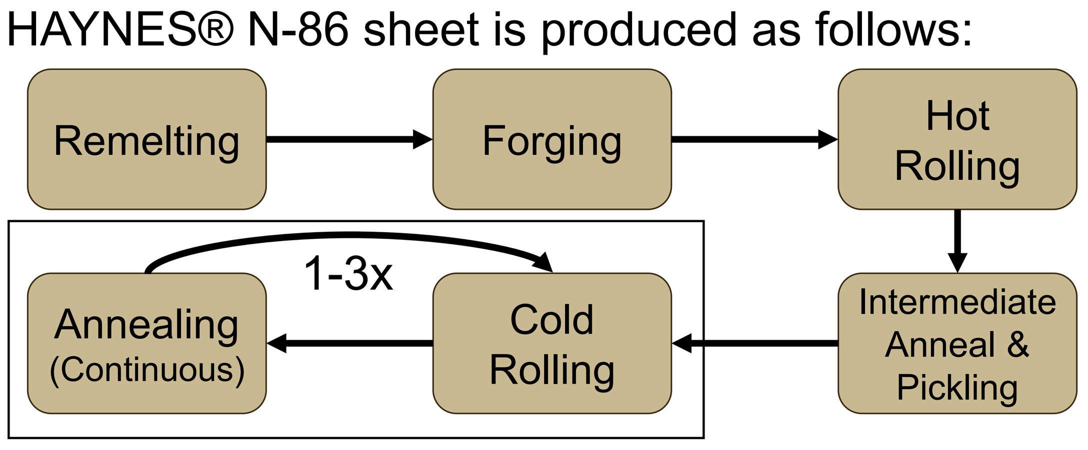
Background

HAYNES® N-86 is a nickel-based alloy with significant additions of chromium and molybdenum. The full composition can be seen below:

	C	Cr	Mo	Mg	Ce	Ni
wt%	0.05	25.0	10.0	0.015	0.03	Rem.

This alloy occasionally fails to meet mechanical property specifications. The goal for this project is to mitigate this issue by defining the relationship between processing parameters and microstructural and mechanical properties.

HAYNES® N-86 sheet is produced as follows:



This research studied the cold rolling and continuous annealing processes (boxed above) used to produce the final sheet thickness and the resulting mechanical properties of HAYNES® N-86.

Experimental Procedures

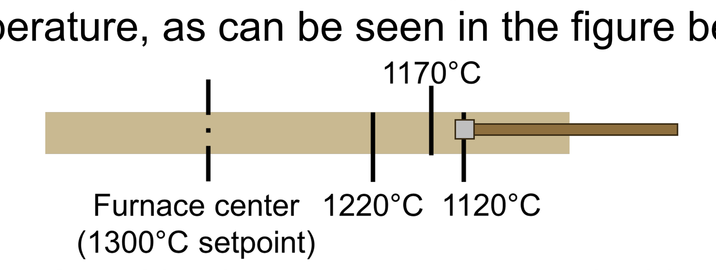
Haynes provided the team with HAYNES® N-86 samples in the pre-cold worked state. To determine the effects of cold work and annealing temperature on hardness and tensile properties, a 12-condition experimental matrix was developed. The anneal time was 2 min for all conditions.

		Cold Work (%)			
		15	30	45	60
Anneal Temp (°C)	1120	C_1	C_2	C_3	C_4
	1170	C_5	C_6	C_7	C_8
	1220	C_9	C_{10}	C_{11}	C_{12}

In addition, an annealing time study was performed to examine the recrystallization behavior and determine the minimum anneal time for complete recrystallization. Samples rolled to 45%CW were annealed at 1170°C for 0, 0.5, 1, 2, and 5 min at temperature.

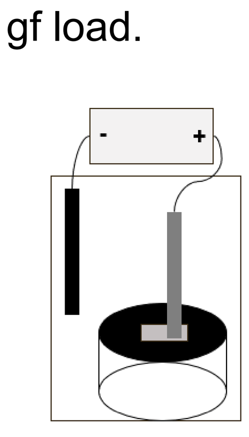
Rolling: Samples were cold rolled in passes of 5-10%CW to the desired overall reduction using a Stanat 2-high rolling mill with 5 in. (127 mm) diameter work rolls.

Short-Time Annealing: The samples were tied to a Type-K thermocouple and placed in a tube furnace. The position of the sample within the furnace was adjusted to achieve the desired temperature, as can be seen in the figure below.



Hardness Testing: Hardness was measured with a Wilson Vickers micro-indentation machine with a 500 gf load.

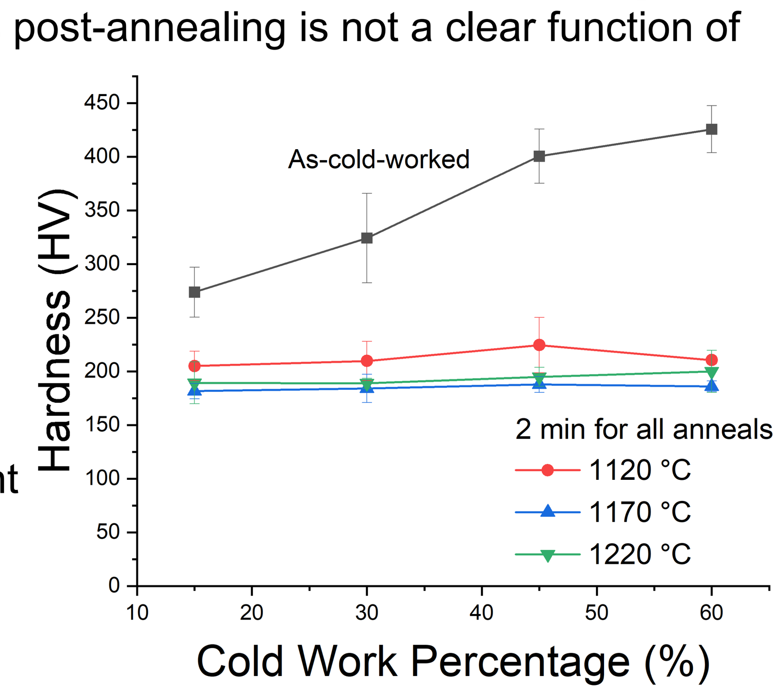
Metallography: Cross-sectional samples were polished up to 800 grit SiC paper and 3-µm diamond paste and then finished with colloidal alumina before electrolytic etching in HCl-5vol%H_2O_2 at 25°C to examine the microstructure via optical microscopy and grain size measurement according to ASTM E112. The etch setup is shown to the right.



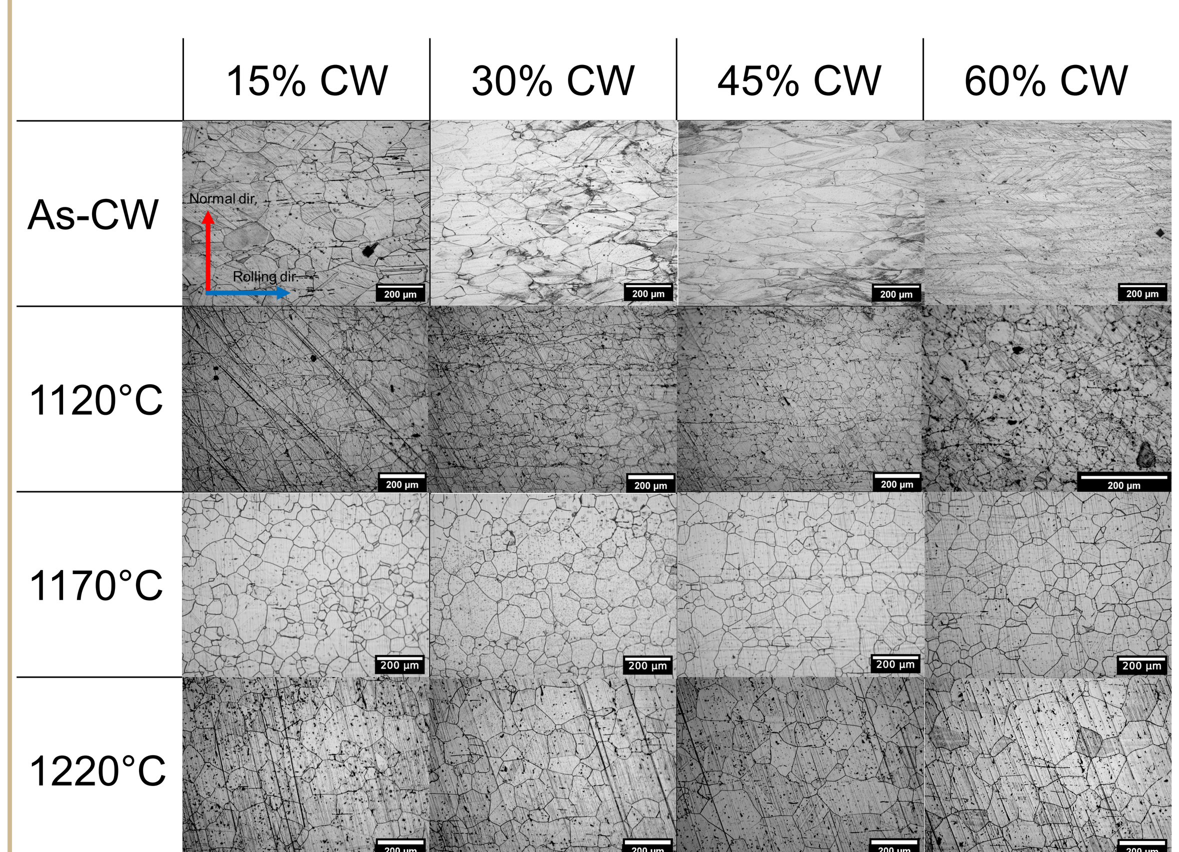
Tensile Testing: Tensile testing was done at Haynes International according to ASTM E8 standard. Tests were done at 21°C (2 samples per condition) and 850°C (1 sample per condition)

Annealing Temperature-Cold Work Study

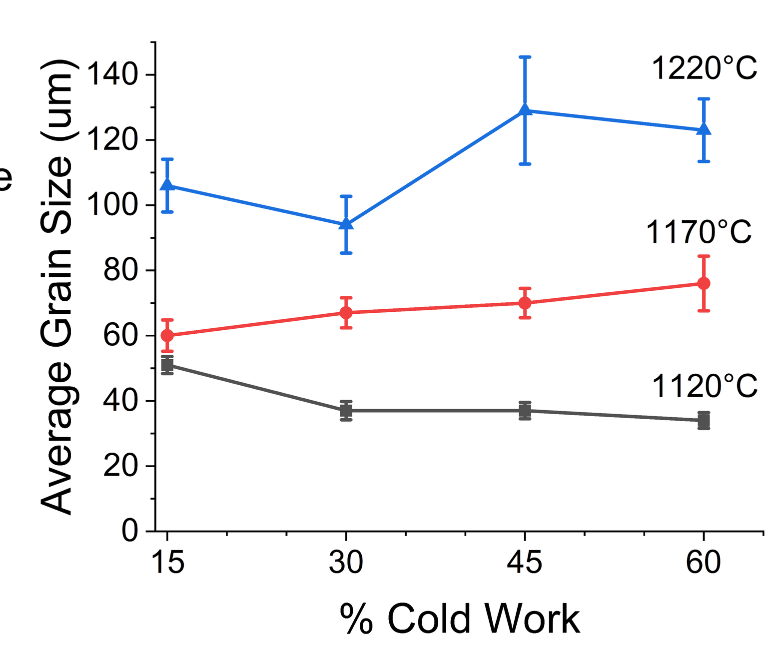
Hardness Testing:
A decrease in hardness was observed after annealing. In addition, hardness post-annealing is not a clear function of prior cold work. Samples annealed at 1120°C did not decrease in hardness as much as 1170°C and 1220°C, possibly indicating those samples underwent incomplete annealing.



Metallography:
The grains were more equiaxed in the annealed conditions than the as-cold worked condition, with large equiaxed grains appearing in the higher anneal temperature samples. The 1120°C anneal resulted in incomplete recrystallization. A significant difference in grain size across different regions of the sample was not apparent.



Grain size measurement confirmed the trends that could be seen visibly in the optical micrographs. The significantly lower grain size in the 1120°C samples is further evidence of incomplete recrystallization.



Tensile Testing:
Tensile properties are mostly as expected for both test temperatures. The 60% CW properties differ from the lower %CW conditions by a significant amount, indicating annealing may not allow for full recovery at such high reductions. Elevated temperature also shows significant differences for 60% CW, although the small sample size is a limitation.

Room Temperature Tests:

1120°C Anneal	30% CW	45% CW	*60% CW*	1170°C Anneal	30% CW	45% CW	*60% CW*
UTS (MPa)	1060	1150	*1510*	UTS (MPa)	958	970	*1210*
YS (MPa)	915	997	*1200*	YS (MPa)	705	505	*1050*
Elongation (%)	22.0	15.2	*10.0*	Elongation (%)	31.8	37.5	*10.0*

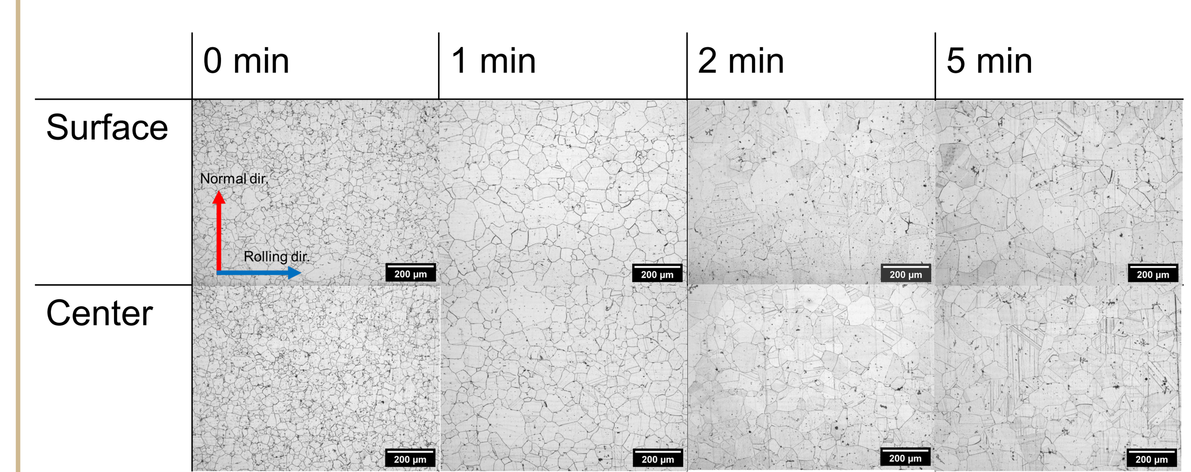
Elevated Temperature Tests (850 °C):

1120°C Anneal	30% CW	45% CW	60% CW	1170°C Anneal	30% CW	45% CW	60% CW
UTS (MPa)	343	333	--	UTS (MPa)	357	332	249
YS (MPa)	231	214	--	YS (MPa)	252	203	168
Elongation (%)	44.0	56.5	--	Elongation (%)	32.0	64.5	102

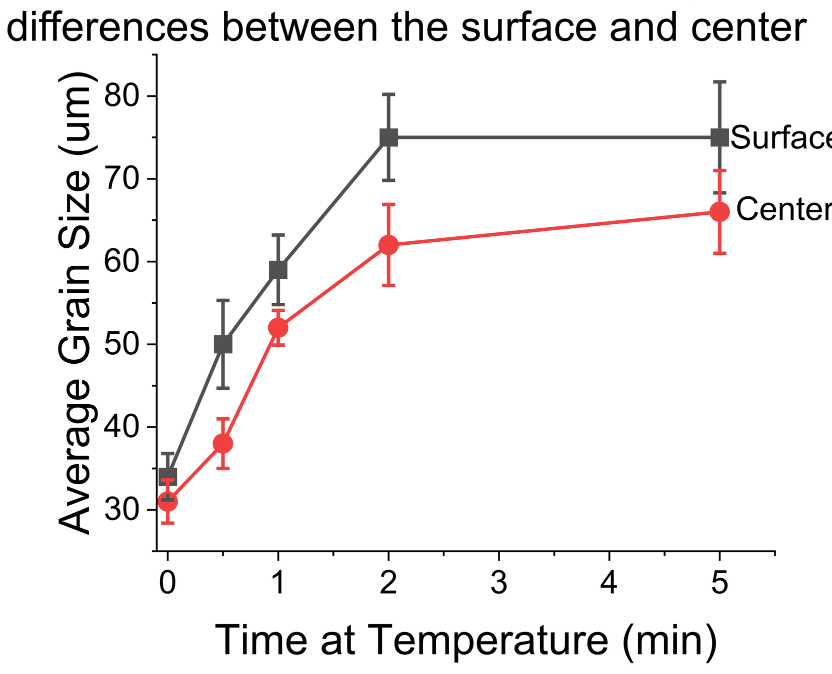
Annealing Time Study

Hardness Testing:
Hardness was approximately 185 HV for all anneal times, indicating minimal change in hardness after initial recrystallization. No significant difference in hardness was measured between the surface and center of any sample.

Metallography:
Grains near the surface of the samples were larger than those near the center of the samples, which could be due to cold rolling mechanics or slow heat transfer to the center during annealing.



Grain size measurements confirmed the qualitatively observed size differences between the surface and center grains. The grain sizes sharply increase up until 2 minutes, with a region of interest between 1 and 2 minutes.



Conclusions and Recommendations

Annealing at 1170°C for 2 minutes was sufficient for acceptable microstructures and properties to be developed. Lowering the anneal temperature should not be considered due to incomplete recrystallization observed at 1120°C. Changes in tensile properties with %CW indicated that a single cold work-anneal cycle should not exceed 45%CW. The annealing time study indicated that annealing time could be lowered due to the recrystallization seen in just 1 min of annealing time. Tensile testing for shorter-time anneals would need to be done to validate this proposal.

Further tensile testing, especially at elevated temperature, needs to be done to properly draw conclusions about the processing effects on mechanical properties. Other variables from earlier in the process (i.e., before cold work) could be investigated for their effects on the final annealed microstructure and properties.

Acknowledgements and References

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