

Haynes International is looking to optimize their rolling process of HASTELLOY® X and HASTELLOY® C-276. The focus is placed on investigating different percentages of cold-working (CW) and annealing times and temperatures to aid in finding the most streamlined processing of the two alloys. Received samples underwent their standard pre-annealing processes. Both alloys underwent 3 rolling sessions, with HASTELLOY® X focusing on varying annealing times and HASTELLOY® C-276 focusing on annealing temperature, while still incorporating both time and temperature changes.

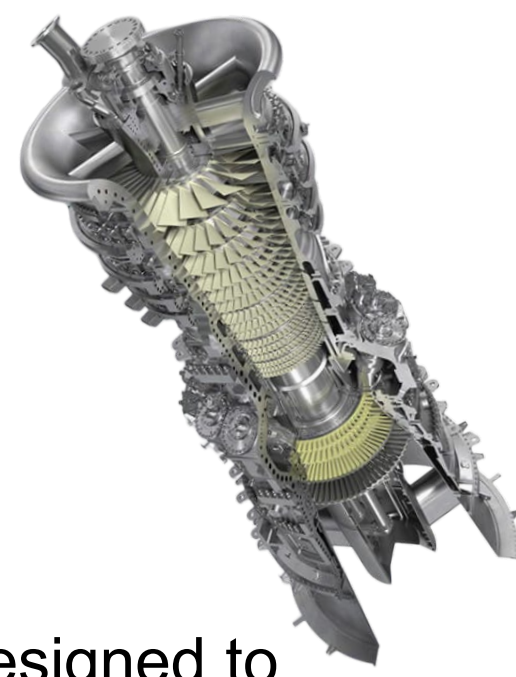
This work is sponsored by Haynes International, Kokomo, IN



Background & Objectives

Background

HASTELLOY® X is a nickel-based superalloy that is solid solution strengthened with a carbide strengthening supplement. It is a jack of all trades alloy with a high oxidation resistance, good strength at high temperatures, and easy to fabricate. This makes it an ideal choice for use in gas-turbine engines for combustion zone components [1].



HASTELLOY® C-276 is a nickel-based superalloy designed to alleviate concerns over welding. It is ductile and very easy to weld, with exceptional resistance to stress corrosion cracking in chloride bearing solutions and sour oilfield environments. It resists both oxidizing and non-oxidizing acids. Often used in chemical processing, oil and gas production, pollution control, and pharmaceutical production [2].

Objectives

Haynes International currently quotes their clients for material costs based on the processing steps and time, along with the bulk material costs. There have been issues where more processing is needed than initially quoted for. Therefore, they are looking to further understand the processing of HASTELLOY® X and HASTELLOY® C-276. Our team is tasked with investigating different cold-working and annealing parameters to aid Haynes' research into whether their processing method for these alloys can be improved.

Alloy Composition

Element	Ni	Cr	Fe	Mo	Co	W	C, Mg, Si, Bo, Nb, Al, Ti, Cu, V
HASTELLOY® X Wt. %	47	22	18	9	1.5	0.6	< 1
HASTELLOY® C-276 Wt. %	57	16	5	16	2.5	4	< 1

Experimental Procedure

Session	HASTELLOY® X			HASTELLOY® C-276		
	Session 1	Session 2	Session 3	Session 1	Session 2	Session 3
66%	3 min @ 2100° F	3 min @ 2100° F	1.5 min @ 2150° F	3 min @ 2075° F	3 min @ 2075° F	1.5 min @ 2075° F
	3 min @ 2150° F	55%	3 min @ 2150° F	10 min @ 2075° F	67%*	10 min @ 2075° F
	10 min @ 2100° F	10 min @ 2100° F	1.5 min @ 2150° F	3 min @ 2100° F	3 min @ 2100° F	1.5 min @ 2075° F
80%	3 min @ 2100° F	3 min @ 2100° F	1.5 min @ 2150° F	3 min @ 2075° F	3 min @ 2100° F	1.5 min @ 2075° F
	5 min @ 2100° F	55%	5 min @ 2100° F	3 min @ 2100° F	67%	3 min @ 2100° F
	10 min @ 2100° F	10 min @ 2100° F	1.5 min @ 2150° F	3 min @ 2125° F	3 min @ 2125° F	1.5 min @ 2075° F

*67% was the target reduction, due to mill limitations the real reductions achieved were limited to 55%

These parameters were chosen to vary the annealing times and temperatures based on practices and ranges provided by Haynes. During annealing, HASTELLOY® X is more time dependent, and HASTELLOY® C-276 is more temperature dependent. After each annealing session, the samples were water quenched. The naming convention used in all figures indicates the alloy and processing path undergone by each sample. The path can be read as follows:

[Alloy][Initial cold work %] - [Annealing temperature] - [Annealing time] - [Stage]
 (Alloys: C = HASTELLOY® C-276, X = HASTELLOY® X) (Stages: S0 = as-received, R# = state after # anneals, AF = after final anneal)

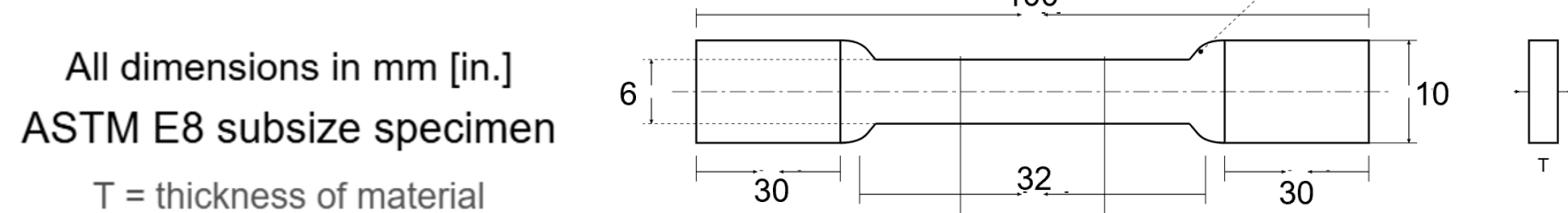
Example: X66 - T2100 - t3 - AF

Hardness Data:

A Wilson Tukon 1202 Hardness Tester was used to collect hardness data for all our test samples.

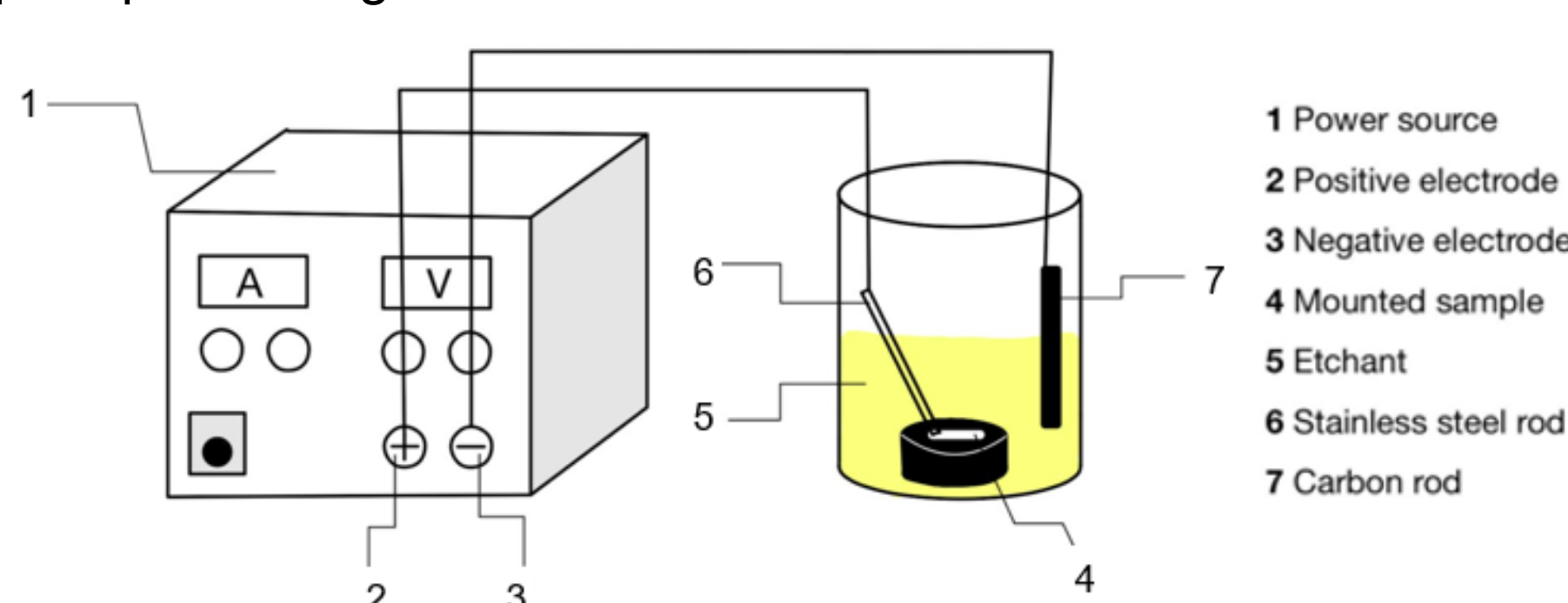
Tensile Testing:

Tested samples were ASTM E8 standard subsize and tests were conducted using a Sintech 30[D] Tensile Tester [3]. A strain rate of 1mm/min was used for all tests. Depending on path, the thickness varied from 0.24 - 3.96mm.



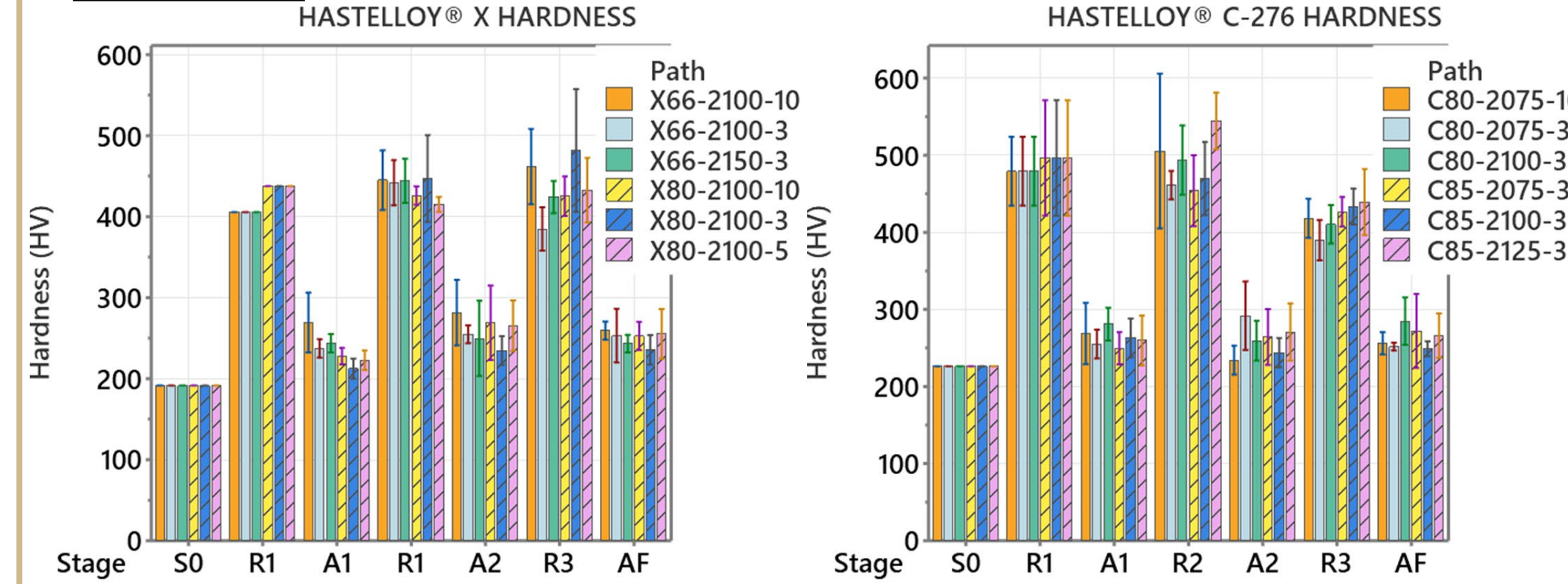
Polishing and Etching Process:

The sample was polished to a 0.05 µm surface finish. The specimen were electrolytically etched in a solution of 95 mL HCl and 5mL 3% hydrogen peroxide at 5 V for ~3-20 seconds varying based on the samples' processing conditions.



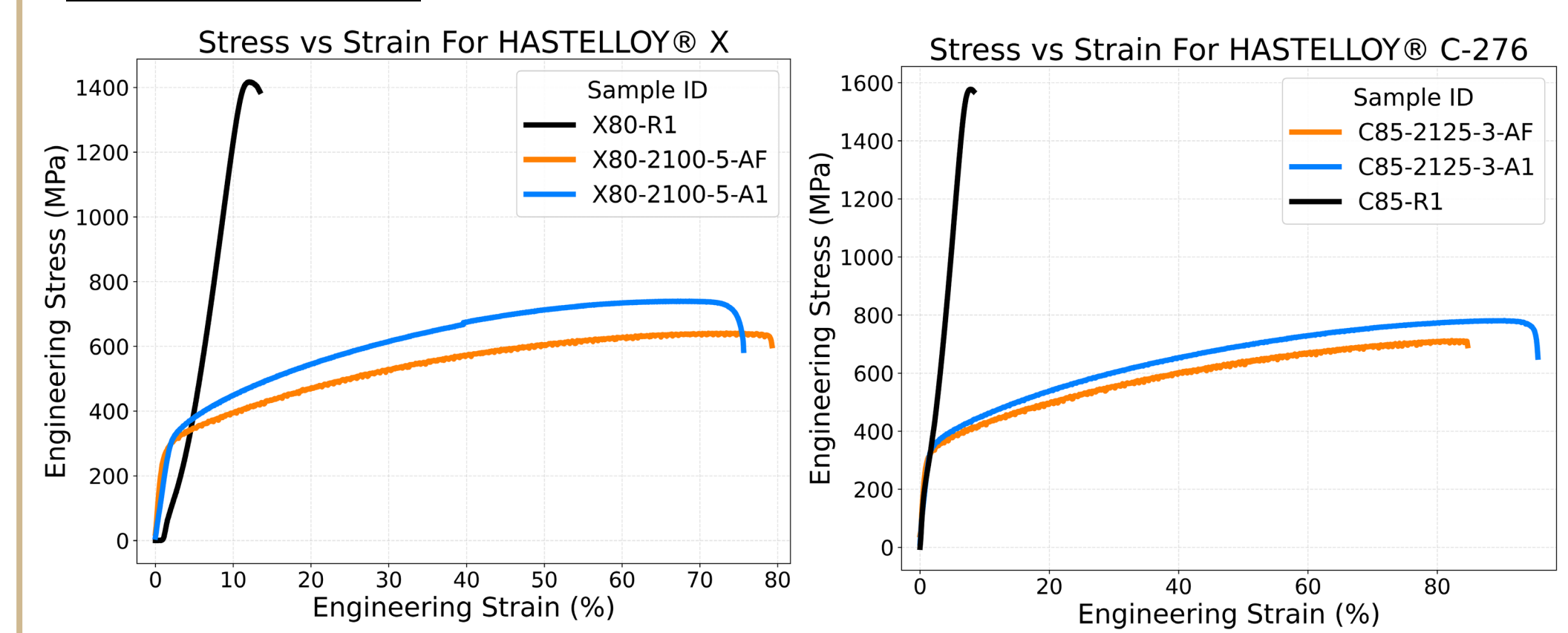
Results

Hardness:

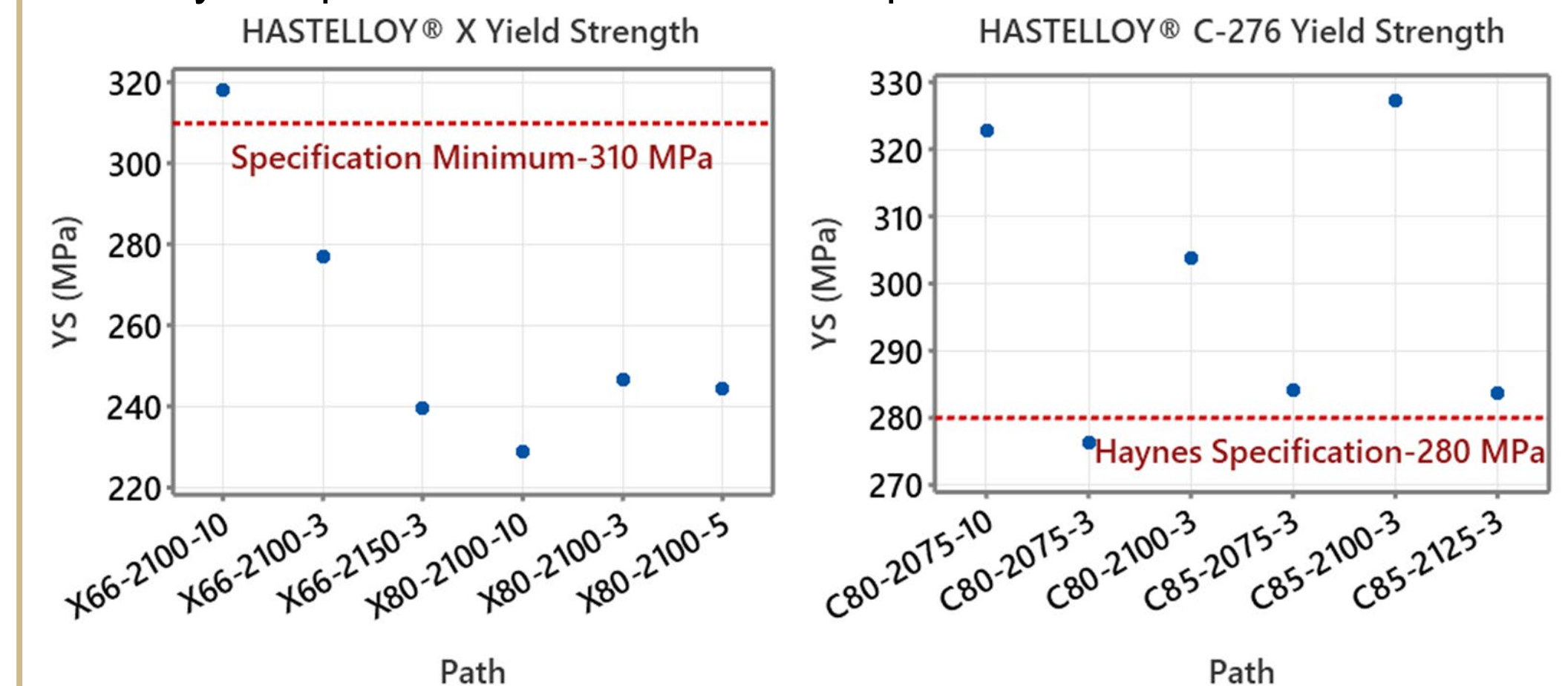


Samples were received at a hardness values of ~200 HV. During rolling it rose to ~400-500 HV and then decreased to ~250 HV after annealing. This reduction is due to successful annealing that reset the crystal structure and reduced hardness to values similar to before cold rolling.

Tensile Testing:

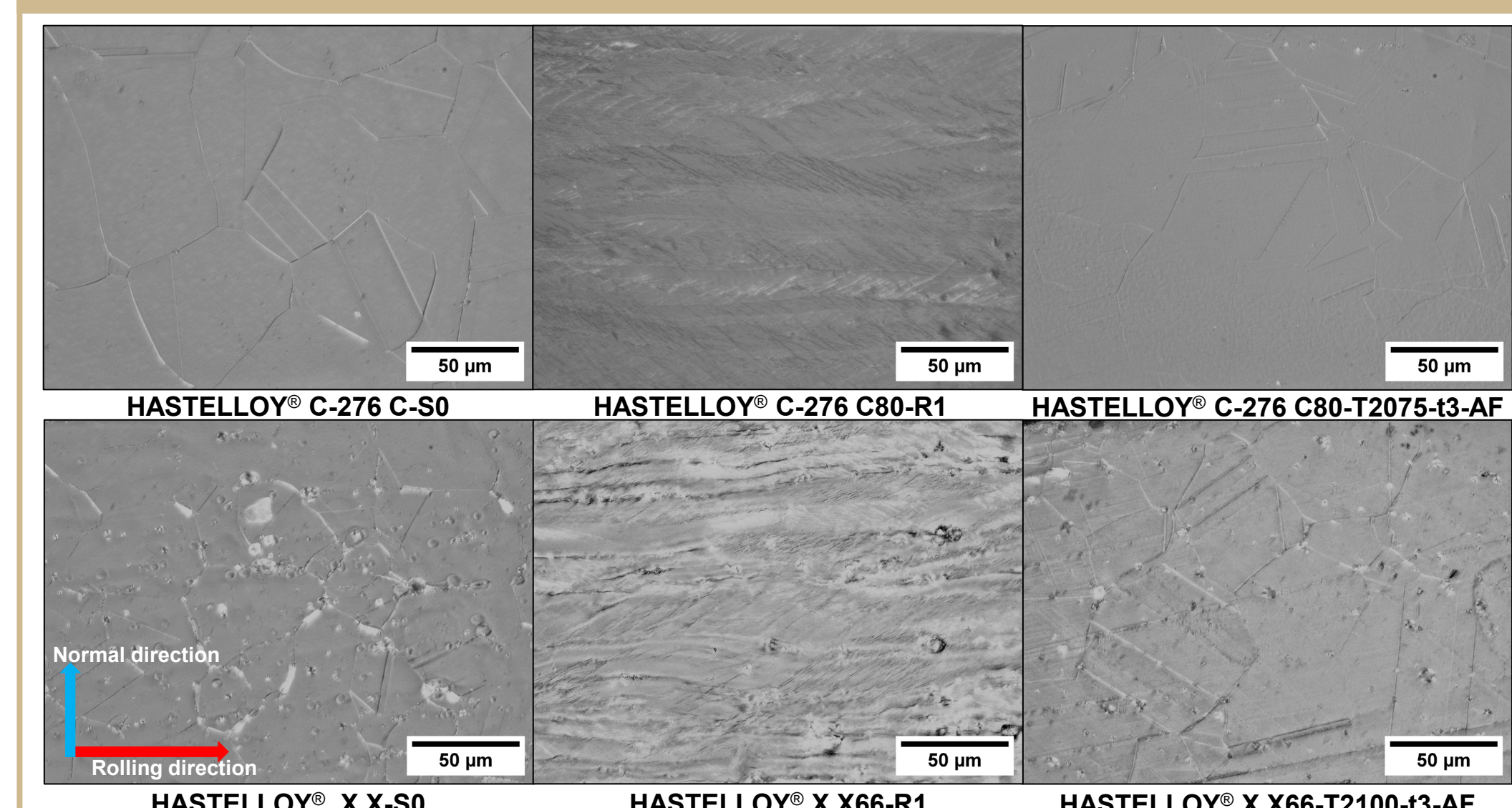


These figures display the influence of three distinct processing stages on their respective mechanical responses. They show the trend of non-annealed samples having a higher UTS with a sacrifice in their ductility compared to the annealed samples.

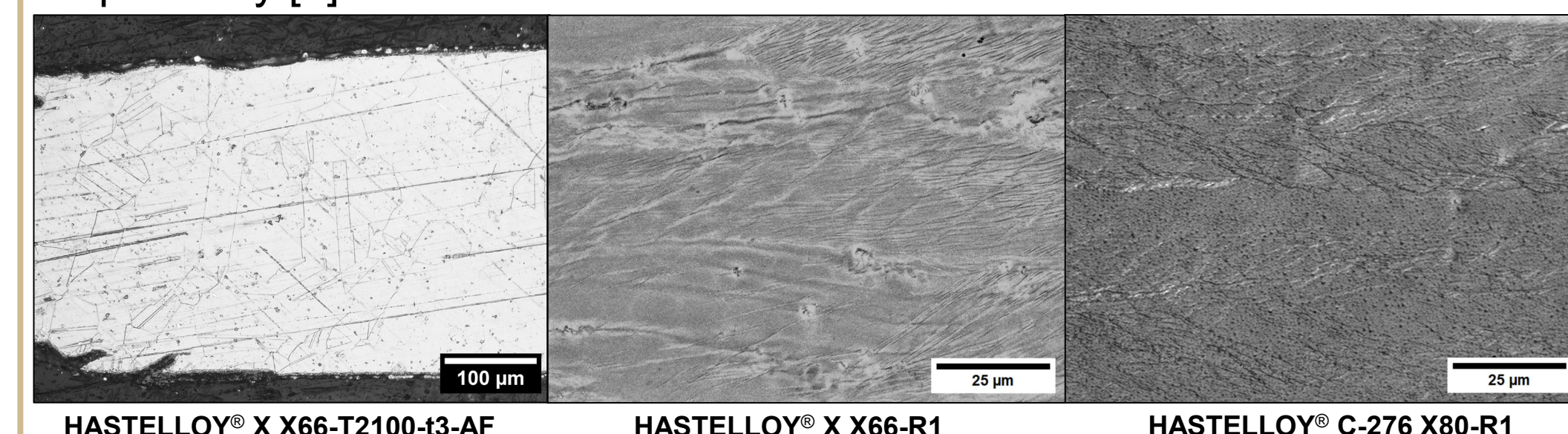


The mechanical properties of the final annealed samples are compared to property specifications for the final property as specified by Haynes. The percent elongation at break was significantly greater than the specifications provided.

Micrographs



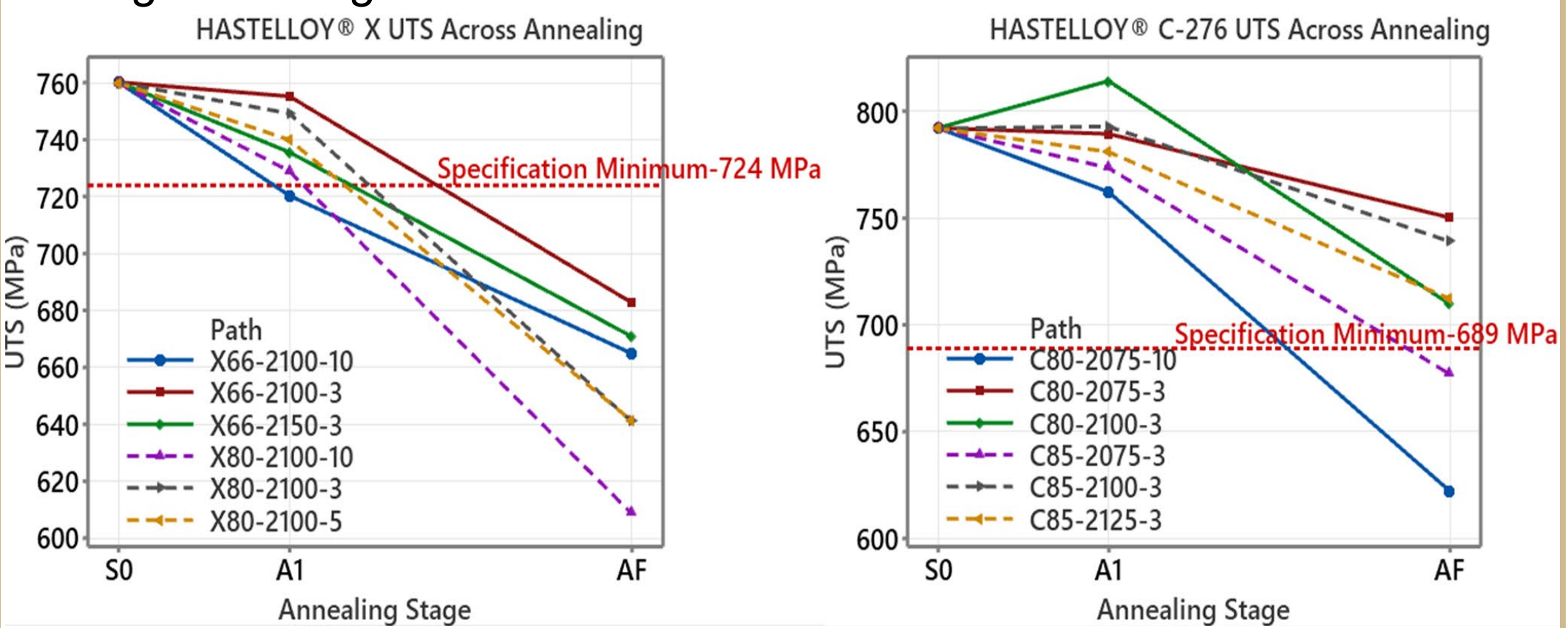
The micrographs above show the microstructures of as-received, rolled and annealed samples of HASTELLOY® C-276 and HASTELLOY® X. Micrographs of cold rolled samples from both alloys show elongated grains as well as slip bands and deformation bands. Micrographs of annealed samples revealed grain recrystallization and increased twinning, showing that the time and temperatures used were sufficient for recrystallization. After annealing, the grain size of both alloys remained much finer than the ASTM 4.0 and 3.0 grain size for HASTELLOY® X and HASTELLOY® C-276 respectively [4].



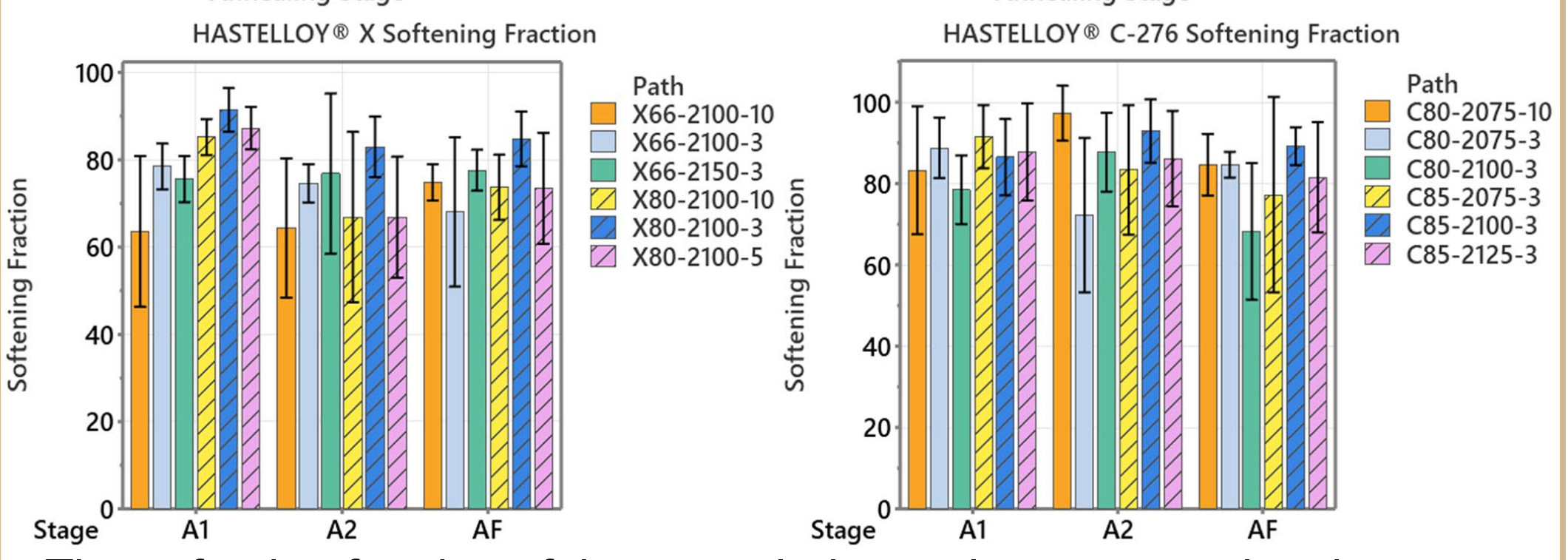
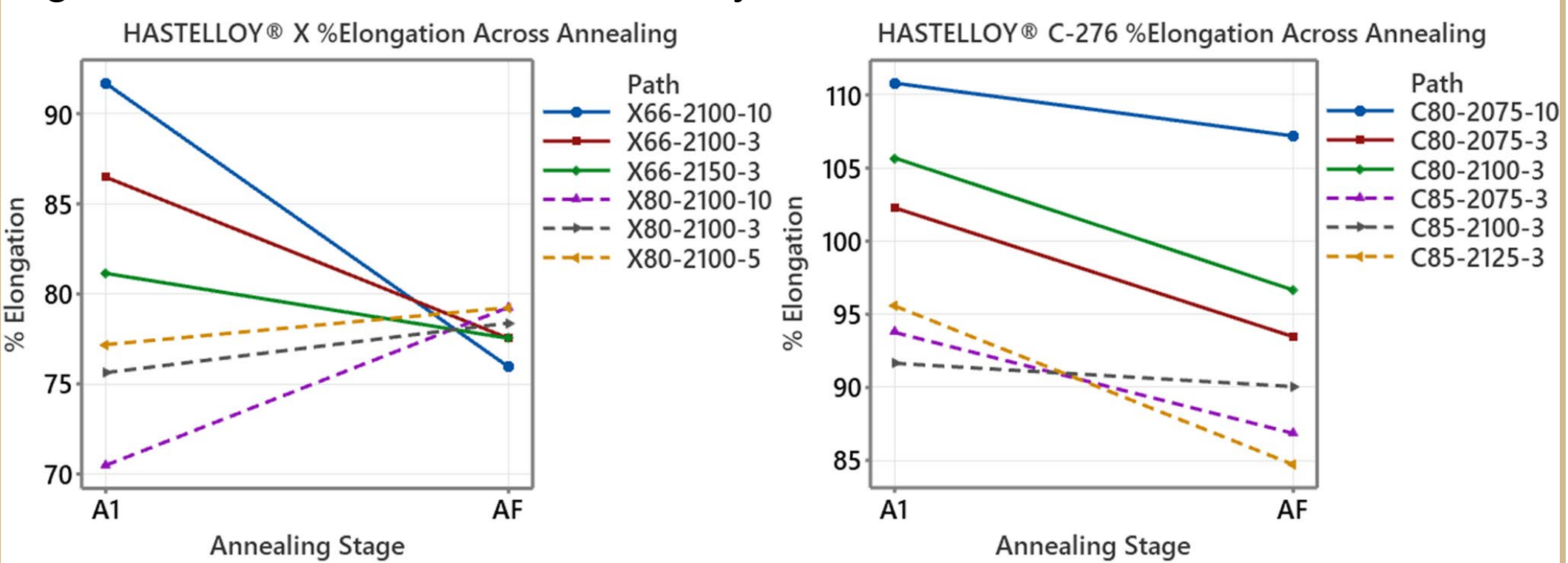
The above micrograph of X66-T2100-t3-AF shows heavy twinning after annealing. The micrographs of X66-R1 and C80-R1 reveal a large amount of slip bands and deformation bands, which is expected [5].

Discussion

The tensile results show the drastic shift in properties between samples that were rolled once (R1) and then annealed (A1). This is due to strain hardening caused by the dislocation density significantly increasing during rolling. Then recrystallization occurs, replacing the grains with high dislocation density with new, strain-free and more equiaxed grains during annealing.



The UTS and % elongation for almost every path decreased after each annealing session. The mechanism that causes this decrease in UTS is relaxation from annealing which allows new strain-free grains to nucleate. These grains grow to consume the deformed microstructure, replacing the elongated dislocation-dense grains with more equiaxed grains with low dislocation density.



The softening fraction of the annealed samples compared to the previous roll was calculated using $\frac{H_{rolled} - H_{annealed}}{H_{rolled} - H_{initial}}$. The softening fraction determined from hardness measurements is directly related to the recrystallization fraction [6]. The HASTELLOY® X sample following the X80-2100-3 path consistently had the greatest softening and there was little variation for the HASTELLOY® C-276 samples.

Conclusions and Recommendations

- For HASTELLOY® X processing, the most optimal parameters for annealing are 2100°F for 3 minutes.
- For HASTELLOY® C-276 processing, the most optimal parameters for annealing are 2100°F for 3 minutes.
- It is recommended that increasing the initial CW% be further investigated due to similar values in the related mechanical properties and grain sizes between different initial CW%. This allows for a potential increase in material yield and process efficiency.
- It is recommended that these tests should be repeated for validity.

Acknowledgements

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