Stainless Steel Microstructure and Mechanical Properties Evaluation

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STAINLESS STEEL MICROSTRUCTURE AND MECHANICAL PROPERTIES EVALUATION

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Abstract

A nitrogen strengthened 21-6-9 stainless steel plate was spinformed into hemispherical test shapes. A battery of laboratory tests was used to characterize the hemispheres. The laboratory tests show that near the pole (axis) of a spinformed hemisphere the yield strength is the lowest because this area endures the least "cold-work" strengthening, i.e., the least deformation. The characterization indicated that stress-relief annealing spinformed stainless steel hemispheres does not degrade mechanical properties. Stress-relief annealing reduces residual stresses while maintaining relatively high mechanical properties. Full annealing completely eliminates residual stresses, but reduces yield strength by about 30%.

Summary

Previous reports KCP-613-8483 and KCP-613-8550 contain the details of the production process for the spinformed 21-6-9 hemispheres. This report contains the data from laboratory testing of the hemispheres. Distortion severity, residual stresses, metallography, microhardness, mechanical properties, and macrohardness were evaluated for the hemispheres in as-spun, stress-relief-annealed and full-annealed conditions. Stress-relief-annealing at 1200 ºF for 1 hour resulted in less distortion than full-annealing at 1900 ºF for 1 hour. Residual stresses were only slightly relieved after 1 hour at 1200 ºF, more relieved after 1 hour at 1300 ºF, and completely eliminated after annealing for 1 hour at 1900 ºF.

Concerning microstructure, the as-spun and stress-relieved hemispheres exhibited elongated grains and slip lines at the waist and at 45º locations but uniform grains near the pole. This implies that the waist and 45º locations endured more cold work and plastic deformation. In the hemisphere annealed at 1900 ºF for 1 hour, the microstructure contained equiaxed grains with straight twin boundaries, indicating complete recrystallization. However, some grain growth had occurred near the pole. Vickers microhardness was highest for the metallographic samples near the waist of the hemisphere.

Mechanical property results revealed that as-spun hemispheres had yield strengths up to 128 kilo-pounds per square inch (ksi) near the waist and as low as 72 ksi at the pole. Stress-relief annealing 1 hour at 1200 ºF and 1 hour at 1300 ºF had no significant effect on mechanical properties, whereas annealing 1 hour at 1900 ºF stabilized yield strength within the range of about 60-80 ksi for the entire hemisphere. Rockwell A Hardness (HRA) testing showed that hardness varies greatly near the pole. Again, the as-spun and stress-relieved hemispheres had nearly matching hardness profiles with an average of about 65 HRA. The fully annealed hemisphere was more uniform but much softer around 52-56 HRA.

Discussion

The spin-forming team is steadily advancing in the processing know-how of spin-forming and the metallurgy of various spin-formed materials. The initial purpose of the spin-forming project was to replace the antiquated hydro-forming process—which requires a special foundation—in the new Kansas City Plant (KCP) facility. Potential customers continue to monitor the progress of spin-forming at the KCP. In addition, KCP engineers are identifying opportunities for spin-forming development or improvement of existing production parts. Pure metal and alloy parts with cylindrical symmetry and wall thicknesses of less than about 0.25 inches are potential candidates for spin-forming. Spin-forming helps to produce near-net parts that reduce subsequent machining steps and/or improve final flow and properties.

In two development orders, the KCP has evaluated spinforming equipment using a hemispherical test shape. Materials in the development orders included 304L stainless steel, 21-6-9 stainless steel, 6061 aluminum, pure copper, pure vanadium, tantalum-2.5% tungsten, and alloy titanium (Ti-6Al-4V). Spinforming successfully produced the final shape for all of the materials except titanium (with a limited number of trials at room and elevated temperatures). The results for the two development orders are presented in reports KCP-613-8483 and KCP-613-8550.

Purpose and Scope

This project evaluated the microstructure and mechanical properties of spinformed 21-6-9 hemispheres in three conditions: as-spun, stress-relief annealed, and fully annealed.

Activity

Stainless steel 21-6-9 plate material with the composition listed in Table 1 was spinformed into a hemispherical shape using two rollers with a single pass program, a feed rate of 800 mm/min, 600 rpm, and added coolant. The starting diameter was 11.65 in and the final diameter was about 8 in. Figure 1 shows the three hemispheres chosen for testing in the as-spun condition. Part 19, 20, and 21 were chosen because they had identical processing parameters and looked nearly identical.

Table 1 Composition of the Annealed 21-6-9 ESR Stainless Steel Plate

19.3% Cr	7.3% Ni	9.35%Mn	.475% Si	.272% N	.0355% C
.0115% Al	.0014% O	.0016% S	.015% P	balance Fe	

A. Part 19

B. Part 20

C. Part 21

Figure 1 **Outer and inner photos of the three 21-6-9 stainless steel hemispheres used for mechanical and microstructural evaluation.**

Heat Treatment

Figure 2 shows the process used to characterize the hemispheres. A circular datum was cut for the purposes of measurement on a coordinate measuring machine (CMM). Also a "skin-pass" machining step was performed to remove only the roughness of the surface so that the hemispheres could be set-up for measurement and residual stress testing. Part 20 was measured on CMM before and after a stress-relief anneal at 1200 ºF for 1 hour in a vacuum furnace. Similarly Part 21 was measured before and after a full anneal at 1900 ºF. Subsequent processing and testing is also shown in Figure 2.

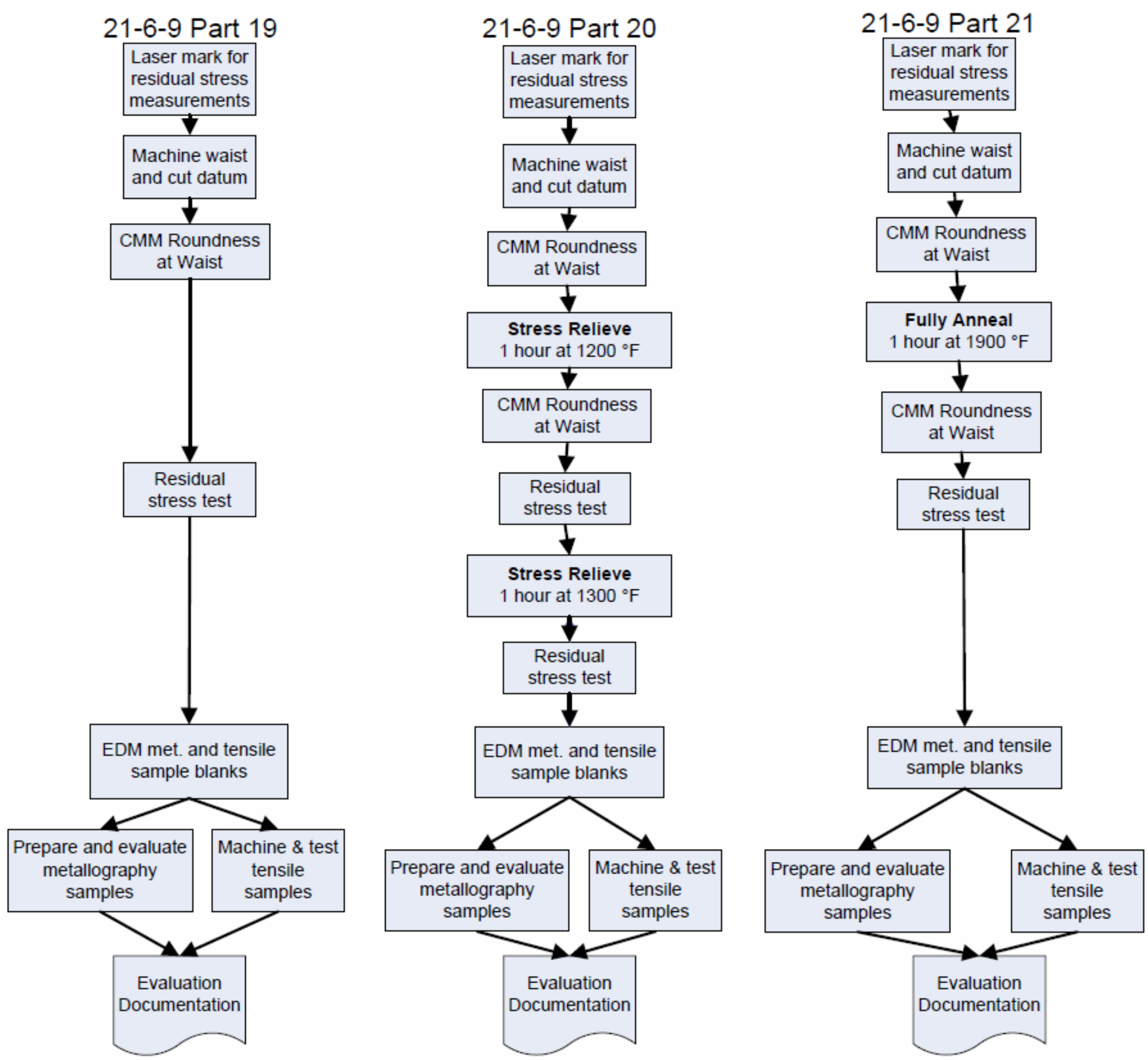


Figure 2 Flowchart for testing of the three 21-6-9 stainless steel hemispheres.

Roundness and Distortion

A Zeiss CMM measured roundness of the 21-6-9 stainless hemispheres by touching over 4500 points with a probe of 0.1574 inches in diameter. Measurements were taken at 0.16 inches above the round waist of the hemisphere. Table 2 lists the roundness measurements before and after heat treating cycles. Part 19 had no subsequent treatment, but Part 20 was measured before and after stress relief for 1 hour at 1200 °F. Part 21 was measured before and after annealing for 1 hour at 1900 °F. In the as-received condition, Part 19 had a poor value for Max Out-of-Round for unknown reasons. What is clear, though, is that the stress-relief-anneal for Part 20 and the full anneal for Part 21 increased the Max Out-of-Round and Standard Deviation values. This is due to shape change as the material relaxes (Part 20) and recrystallizes (Part 21). Observe that the stress relief anneal at 1200 ºF for one hour resulted in 0.0030 in Max Out-of-Round, which is less than half the distortion of the full anneal at 1900 ºF, 0.0077 in Max Out-of-Round.

Table 2 Roundness and Distortion of the 21-6-9 Stainless Steel Hemispheres

	Average Diameter (inches)	Max Out-of-Round (inches)*	Standard Deviation (inches)	Number of Points
Part 19 as received	9.0391	0.0041	0.0012	4549
Part 20 as received	9.0669	0.0017	0.0005	4544
Part 20 stress relieved at 1200 ºF for 1 hour	9.0680	0.0030	0.0010	4554
Part 21 as received	9.0398	0.0019	0.0005	4547
Part 21 annealed at 1900 ºF for 1 hour	9.0391	0.0077	0.0025	4551

** "Max Out-of-Round" is the difference between the maximum and the minimum points in the CMM data when compared to a perfect circle of the same nominal diameter.*

Figure 3 shows the roundness charts for Parts 19, 20, and 21. The charts show an expanded scale in which the nominal concentric circle shows the average diameter, and the inner and outer circles show the +0.005 inch and -0.005 inch tolerances, respectively. The line trace that deviates from the nominal concentric circle represents the measured distortion. Figures 3A, 3B, and 3D are the as-received hemispheres. Figure 3B was then stress relief annealed at 1200 ºF for 1 hour and distorted slightly as shown in Figure 3C. Figure 3D was fully annealed at 1900 ºF for 1 hour and consequently showed more distortion in Figure 3E.

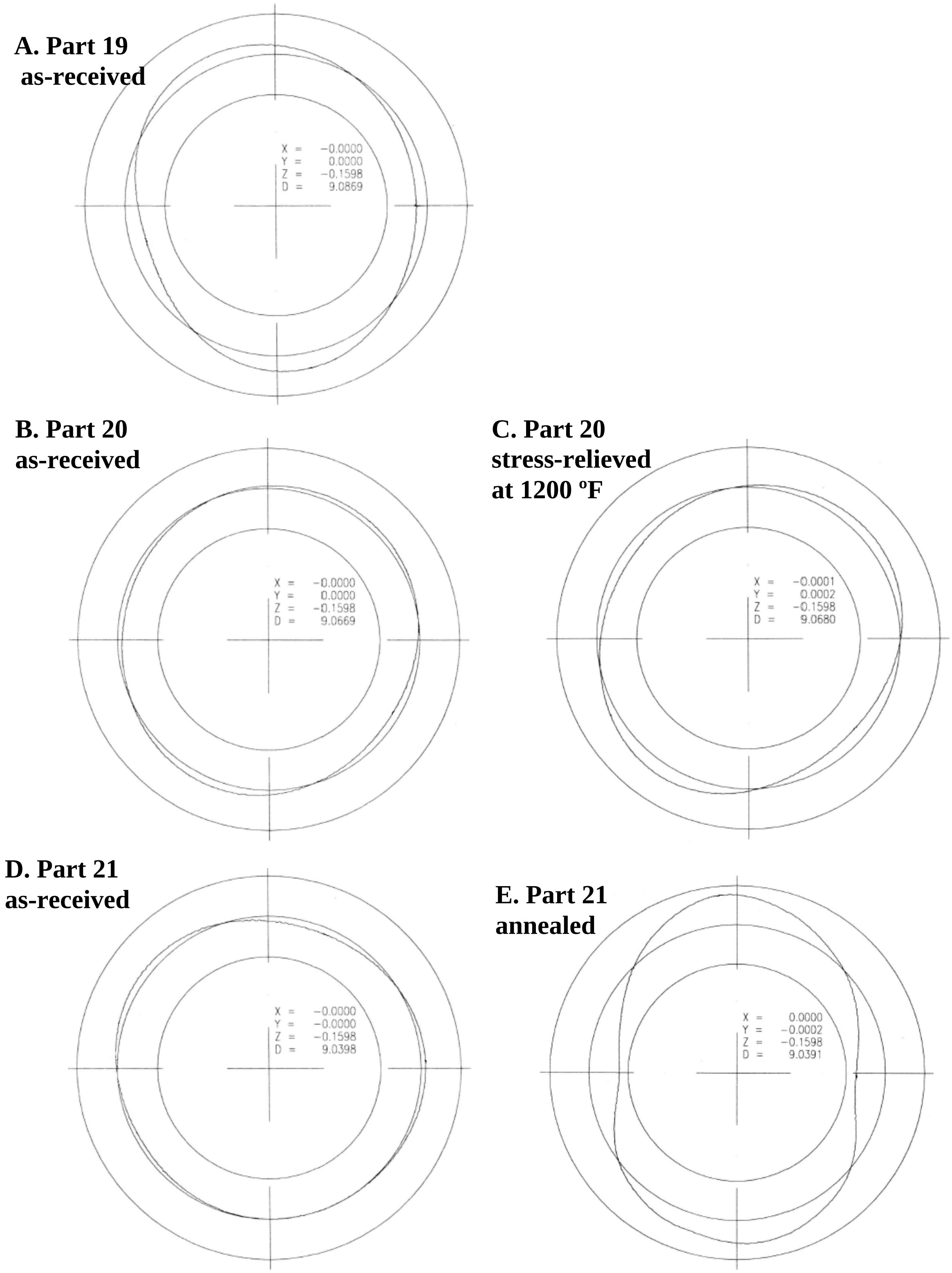


Figure 3 **Roundness charts for 21-6-9 stainless steel hemispheres. Diagrams A-E show an expanded scale in which the nominal circle shows the average diameter, and the inner and outer circles show the +0.005 inch and -0.005 inch tolerances, respectively.**

Residual Stress Analysis

Figure 4 shows the locations of the strain gage rosettes (the gold squares) for measurement of residual stresses on the spinformed 21-6-9 hemispheres. Measurements were made at 0º (pole), 45º, and 67.5º (near the waist). Measurements Group EA-06-062RE-120 gages were attached using M-Bond 610 with a 300 ºF cure. Max stress was measured clockwise from Gage 1 with the following assumed values: Modulus of Elasticity = E =$27.6\text{x}10^6$ psi and Poisson's ratio = v = 0.28. Also visible is the residual epoxy in the locations where the feet of the drill assembly had been glued to the work piece. Figure 5 is a picture of the drill setup and fixture for residual stress measurement. H-Drill software was used in the analysis based on uniform stress theory (principal stresses). The drill diameter was 0.070 inches and the max drill depth was 0.080 inches.

Figure 4 **Residual stress test locations after testing. The bronze-colored strain rosettes are visible at 0 º (Pole location), 45 º, and 67.5 º (Near waist). The holes for the test were drilled in the center of each strain rosette. Leftover epoxy is visible where the feet for the drill press had been glued to the hemisphere.**

Figure 5 **Residual stress test setup for 21-6-9 stainless steel hemispheres. The drill press is centered on the strain rosette and the feet are glued in place on the work piece.**

Table 3 presents the raw data from residual stress measurements. The graphical results in Figure 6 show clearly that annealing for 1 hour at 1900 ºF reduces the magnitude of residual stresses to nearly zero. Due to the drill setup, all the residual stress measurements were made on the outer surface of the hemispheres. On the outer surface the maximum and minimum stresses were mostly compressive, i.e. negative. The highest shear stress was in the location near the waist. Observe that each stress relief heat treatment resulted in a slight reduction in shear stresses at every location on the hemisphere.

Table 3 Residual Stress Measurements of 21-6-9 Stainless Steel Hemispheres

	Location 1	*Location 2*	*Location 3*	*Location 4*
As Spun	*0-deg*	*45-deg*	*67.5-deg 1*	*67.5-deg 2*
Stress max	-15.5	0.7	14.5	10.8
Stress min	-25.7	-32	-30.5	-36.4
Shear max	5.1	16.3	22.5	23.6
Beta (deg)	-45	-89	-90	84
	Location 1	*Location 2*	*Location 3*	*Location 4*
Stress Relieved 1200	*0-deg*	*45-deg*	*67.5-deg 1**	*67.5-deg 2*
Stress max	-29.6	-9.6	4.1	11
Stress min	-33.5	-20.5	-19.9	-30.2
Shear max	2	5.5	12	20.6
Beta (deg)	8	-82	89	-87
			Location 5	
Stress Relieved 1300			*67.5-deg 3*	
Stress max			10	
Stress min			-18.1	
Shear max			14	
Beta (deg)			-87	
	Location 1	*Location 2*	*Location 3*	*Location 4*
Annealed	*0-deg*	*45-deg*	*67.5-deg 1*	*67.5-deg 2*
Stress max	4.4	-2.6	4.1	-2.6
Stress min	2	-4.9	-0.4	-5.7
Shear max	1.2	1.2	2.2	1.5
Beta (deg)	-57	-41	-42	44

** Possible test error due to erroneous drilling depth from gage malfunction.*

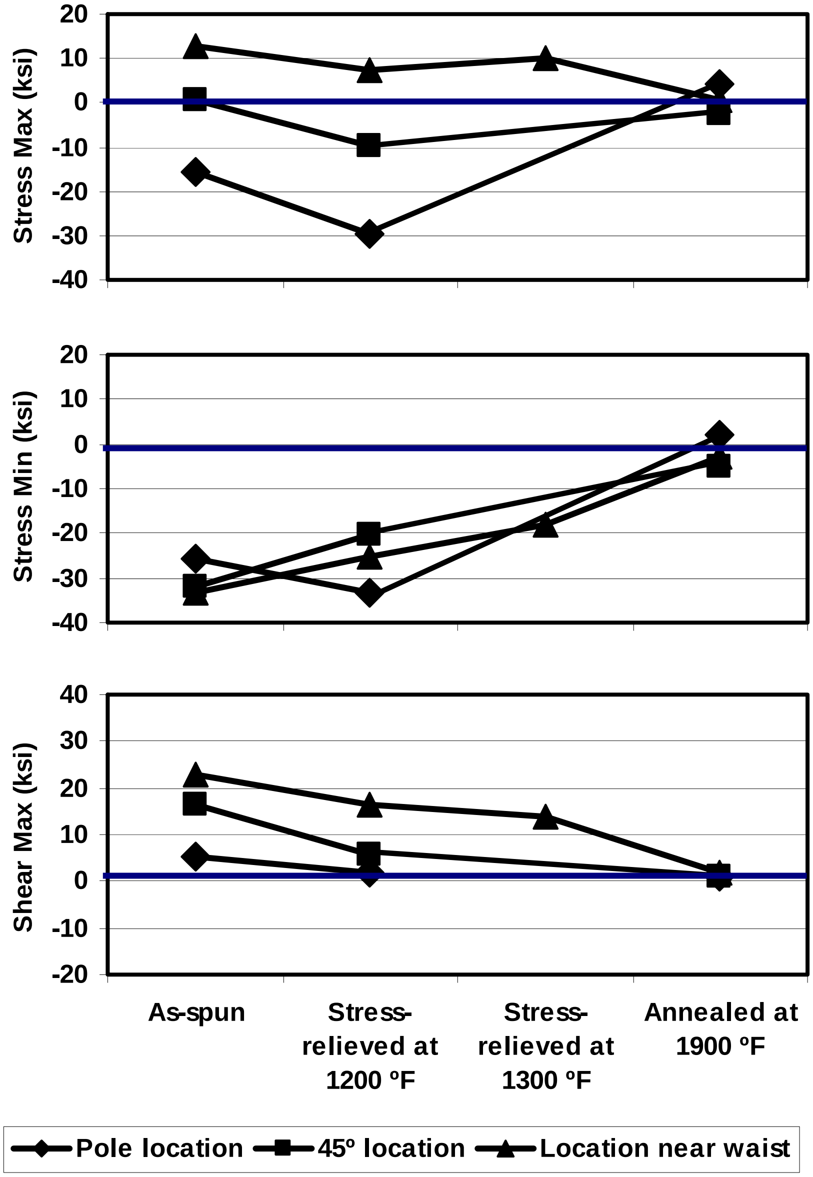
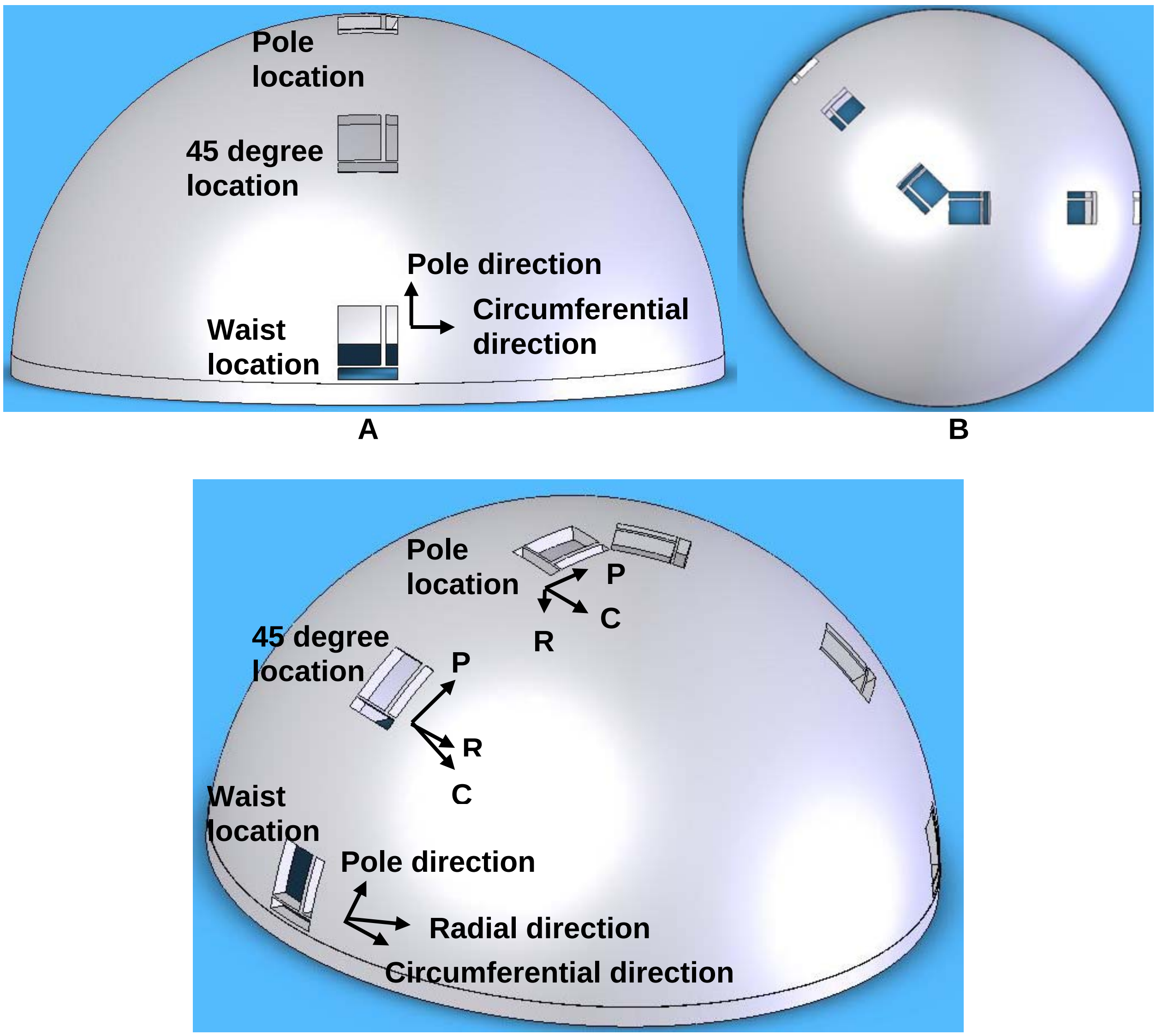


Figure 6 **Outside residual stress measurement results for spinformed 21-6-9 hemispheres. Residual stresses are high in the waist and 45° locations for the as-spun and stress relieved hemispheres. Annealing reduces the magnitude of residual stresses to nearly zero.**

Metallographic Characterization

After residual stress measurement, metallographic samples and tensile test samples were extracted from the spinformed hemispheres using electro-discharge machining (EDM). Figure 7 reveals the locations and orientations of samples removed from hemispheres 19, 20, and 21.



C

Figure 7 **Locations of metallographic (hardness) and tensile samples for spinformed 21-6-9 stainless steel hemispheres. A) Side view, B) top view, and C) trimetric view. Large cut-out rectangles represent metallographic and hardness samples. Slender rectangles represent tensile bar locations. The coordinate system to identify pole, radial, and circumferential directions is shown.**

Figure 8 shows the microstructure of the 21-6-9 annealed plate prior to spinforming. The grains are equiaxed and uniform with a typical grain diameter of about .002 inches (50 µm), and therefore a nominal ASTM grain size of about 5.5. The horizontal shadows in the metallographic image represent flow lines due to the rolling process.

From the spinformed hemispheres, metallographic samples were cut so that microstructure could be observed in all three directions—pole direction, radial direction, and circumferential direction—as specified in Figure 7. Samples were epoxy mounted, polished, and etched with electrolytic nitric acid etch (70% nitric acid) at about 1.1 volt. The etching time varied from 30 seconds to 10 minutes depending on the amount of cold work in the sample. Images were captured at 100X magnification and 200X magnification, but, due to the fine grain size, only the 200X magnification images will be presented in this report.

Figure 9 compares the as-spun, stress-relieved, and annealed microstructures near the waist of each hemisphere. The stress-relief eliminated slip lines due to cold work and the full anneal resulted in recrystalization of the microstructure. Figure 10 reveals that microstructural deformation is more visible near the waist than at the 45º location or near the pole. Near the waist the grains are elongated in the direction of deformation. Figure 11 shows that irregular grain growth occurred near the pole for the hemisphere that was fully annealed at 1900 ºF for 1 hour. This grain growth may have been because less cold work took place near the pole during spinforming compared to the higher amount of cold work that took place at the waist and 45º locations.

Figures 12 and 13 show the microstructure near the waist from three directions: circumferential, pole, and radial. The as-spun sample in Figure 12 does not reveal too much of a difference in microstructure with respect to perspective. However, the stress-relieved sample in Figure 13 shows highly elongated grains in the pole view and somewhat elongated grains in the radial view. The grains in the circumferential view are more or less equiaxed. This implies that the most concentrated deformation occurred in the circumferential direction.

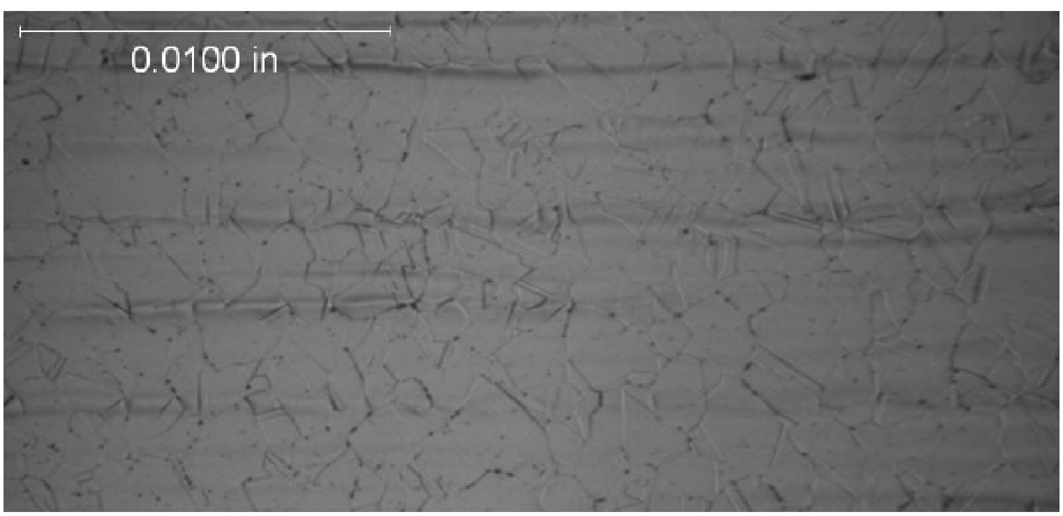
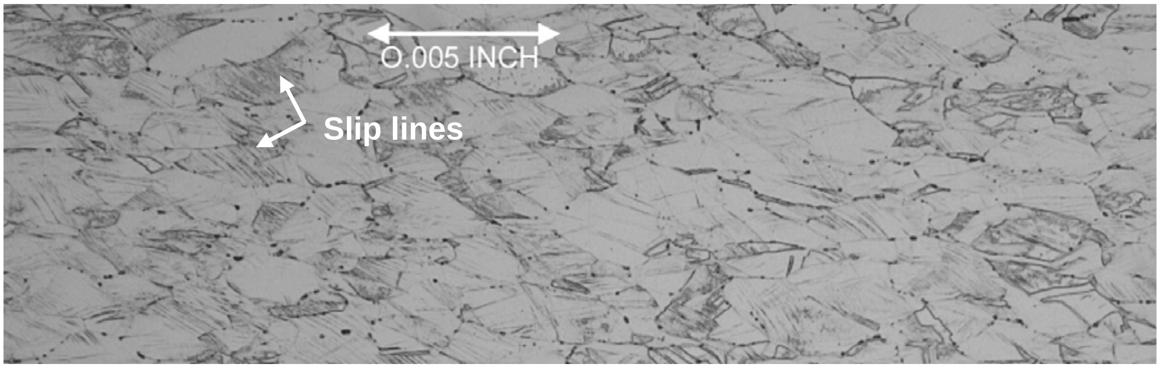
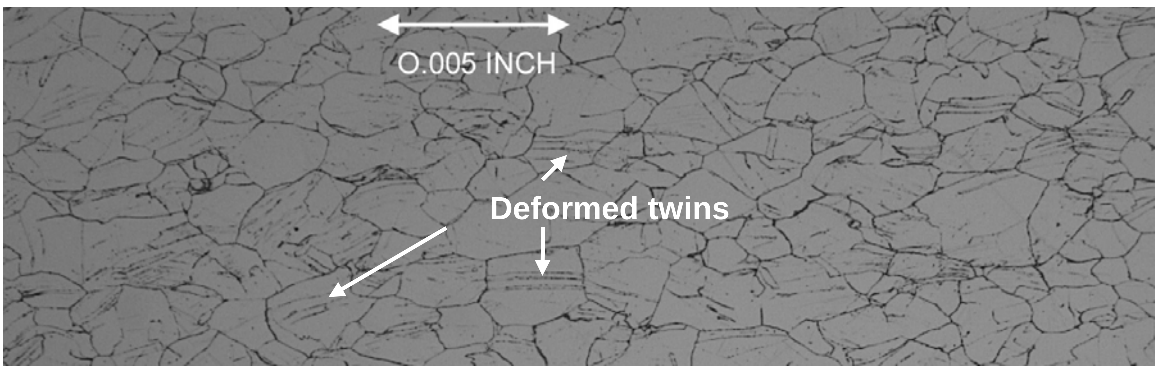


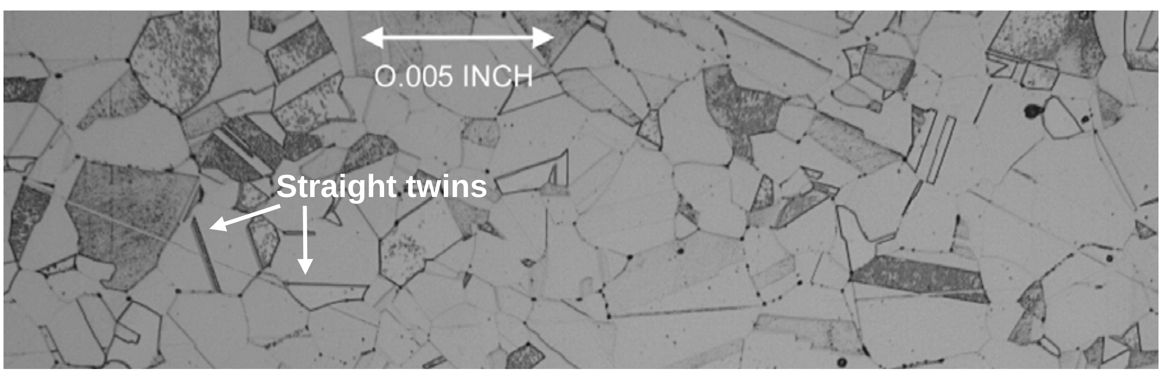
Figure 8 Equiaxed grains in the annealed 21-6-9 stainless plate prior to spinforming.



A (as-spun)

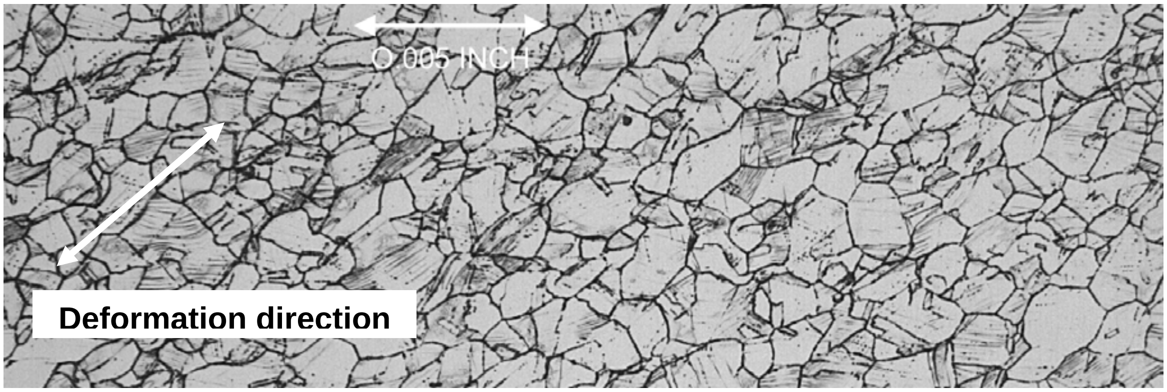


B (stress-relieved)

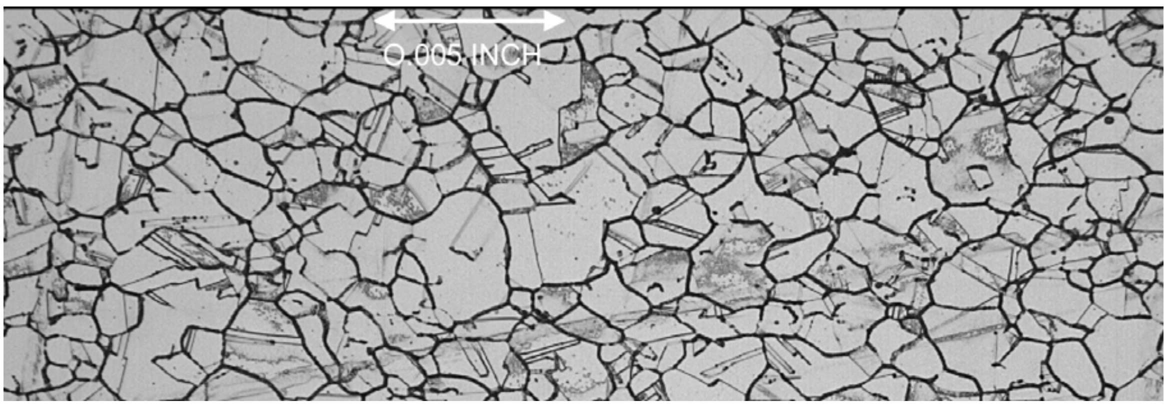


C (fully annealed)

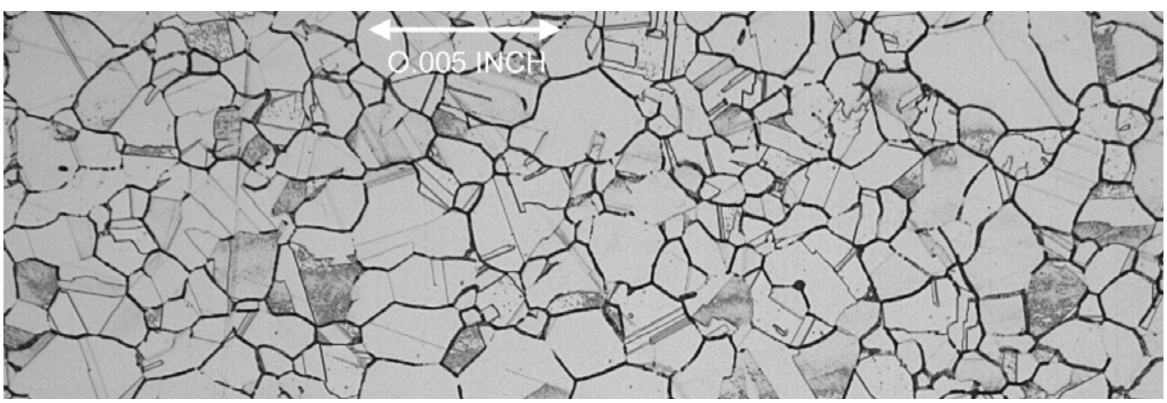
Figure 9 **Effect of heat treatments on the microstructure of 21-6-9 stainless steel spinformed hemispheres: A) As-spun, B) stress-relieved 1 hour at 1200 ºF and 1 hour at 1300 ºF, and C) fully annealed 1 hour at 1900 ºF. The location for all three samples was near the waist, oriented in the radial direction as defined in Figure 7. Note the fine, feathery slip lines in the as-spun sample (A). In the stress-relieved sample (B), the grain boundaries are resolved and deformed annealing twins are more visible. The microstructure has experienced recovery through rearrangement of dislocations to reduce internal energy. In the fully annealed sample (C), recrystallization has occurred as signified by the equiaxed grain structure and perfectly straight annealing twins.**



A (near the waist)

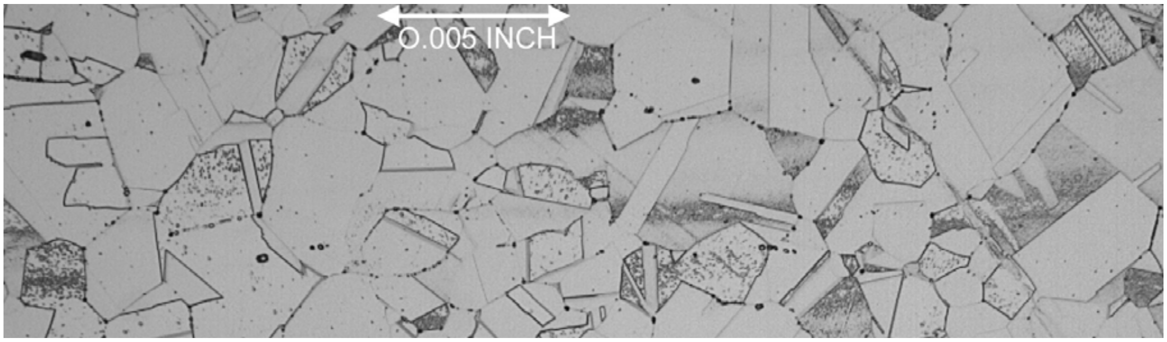


B (at the 45º location)

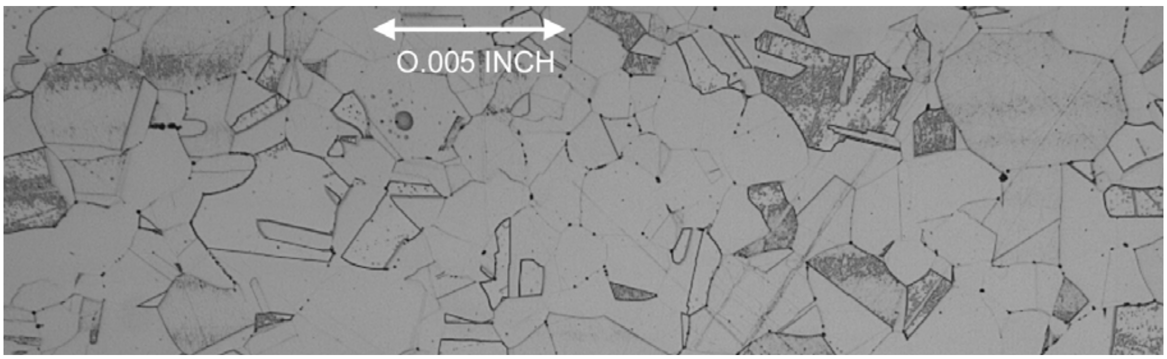


C (near the pole)

Figure 10 Microstructural variation by location within the stress-relieved 21-6-9 stainless steel spinformed hemisphere: A) near the waist, B) at the 45º location, and C) near the pole. All images show the view from the circumferential direction as defined in Figure 7. Notice the directionality of the elongation near the waist (A). At the 45º location (B) and near the pole (C), the grains are relatively equiaxed. Therefore, deformation appears to have been relatively uniform in both the radial and pole direction (as observed in this image of the circumferential view).



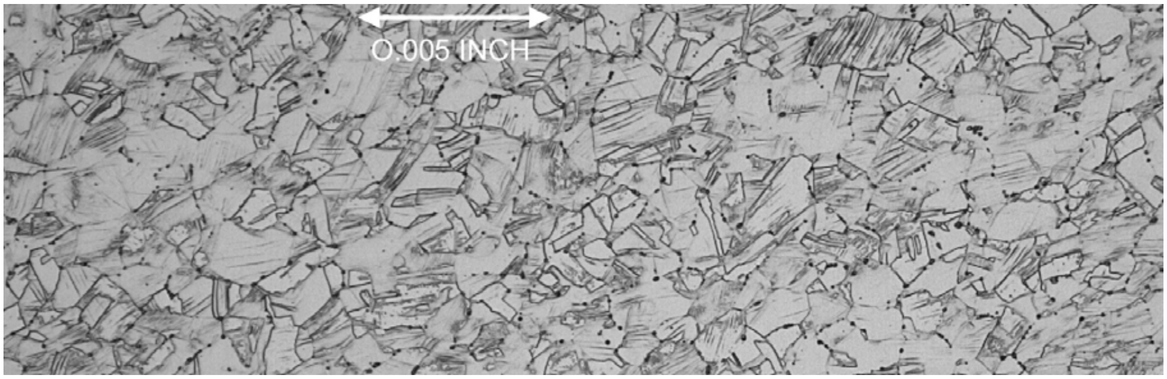
A (near the waist)



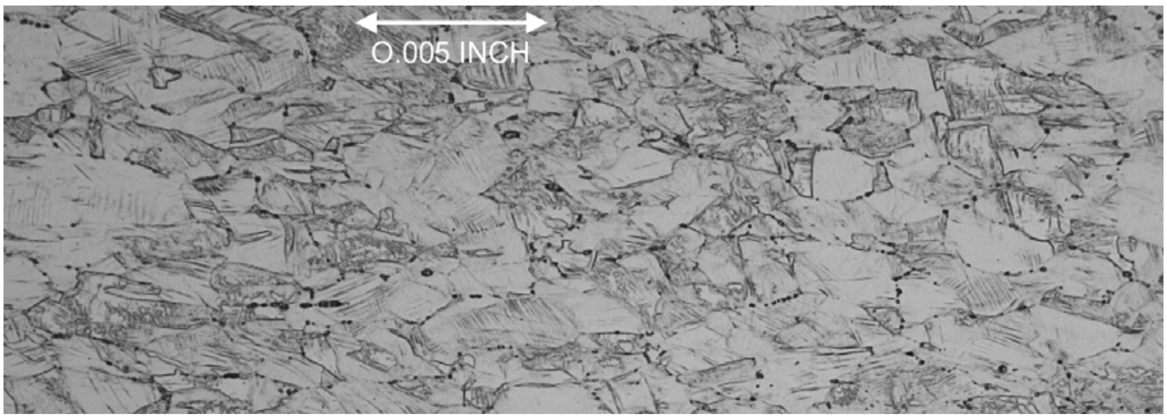
B (at the 45º location)

C (near the pole)

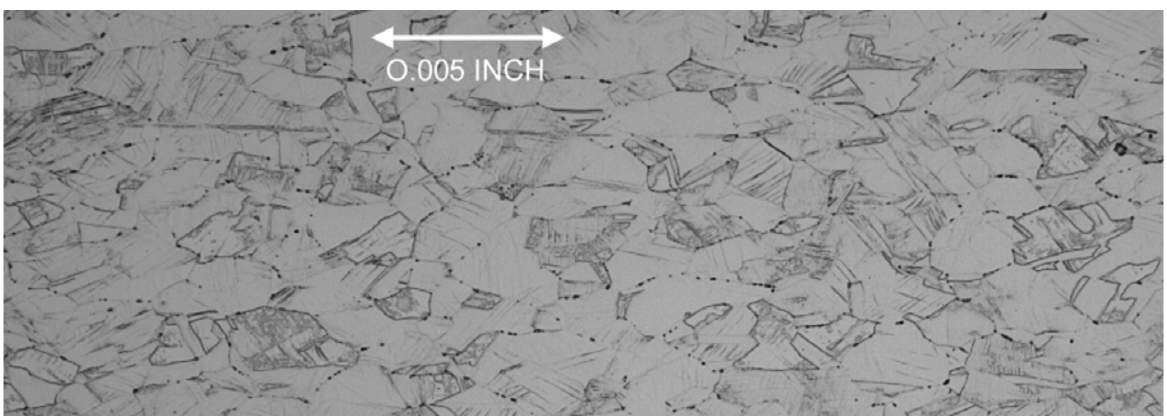
Figure 11 Microstructural variation by location for the <u>fully annealed</u> 21-6-9 stainless steel spinformed hemisphere. A) Near the waist, B) at the 45º location, and C) near the pole. All images show the pole view as defined in Figure 7. The waist and 45º locations (A&B) exhibit relatively small, equiaxed recrystallized grains. However, near the pole, some of the recrystallized grains are large, indicating that grain growth occurred during anneal.



A (circumferential view)

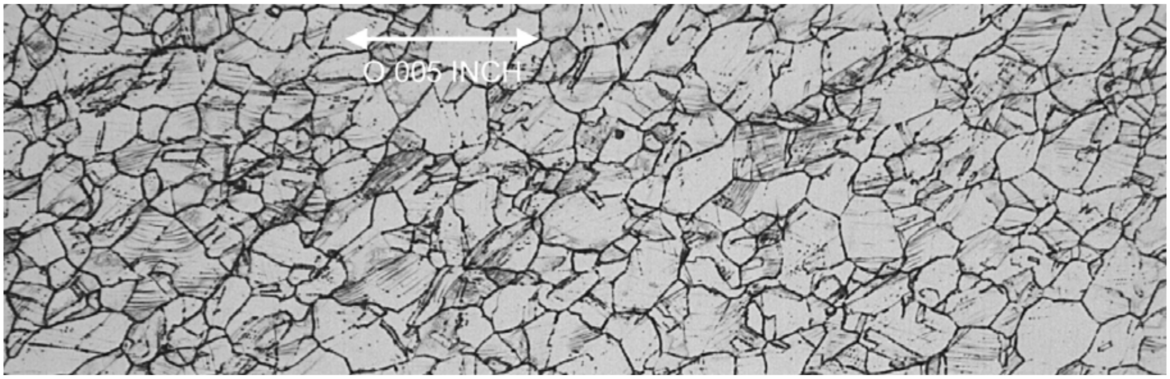


B (pole view)

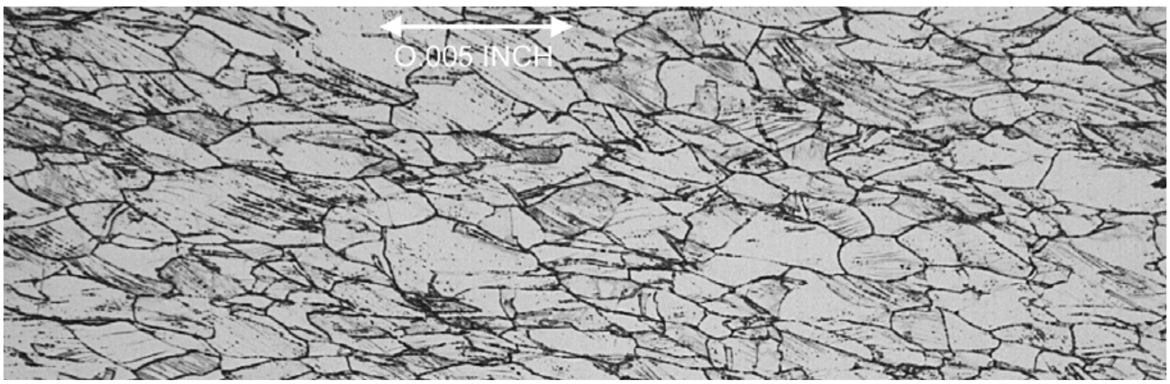


C (radial view)

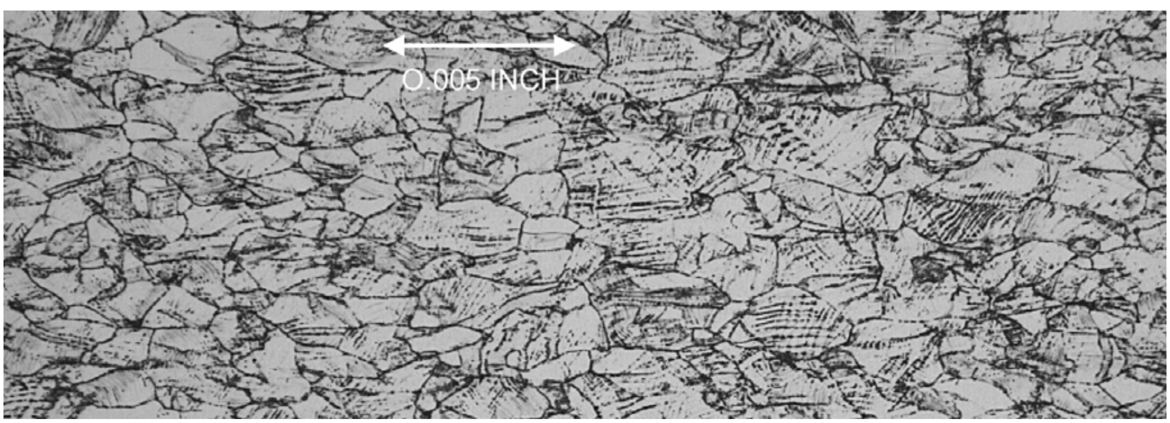
Figure 12 View of the microstructure from all three directions in <u>as-spun</u> 21-6-9 stainless steel spinformed hemispheres. As seen in these metallographic images from samples <u>near the waist</u> there is little difference in microstructure from each of the three perspectives. Slip lines and a slight elongation in the direction of deformation are visible in all three micrographs.



A (circumferential view)



B (pole view)



C (radial view)

Figure 13 Appearance of the microstructure from all three views in 21-6-9 stainless steel spinformed hemispheres stress-relieved 1 hour at 1200 ºF and 1 hour at 1300 ºF. These stress-relieved samples near the waist reveal a greater difference in microstructure due to perspective. Elongated grains are most pronounced in the pole view (B) and to a lesser extent in the radial view, identifying the circumferential direction as the direction of the most concentrated deformation.

Microhardness

Table 4 gives the average of three Vickers 500 g microhardness readings for each metallographic sample. Figure 14 graphs the data for the three spinformed hemispheres with respect to the three locations and three viewing directions. The as-received and stress-relieved hemispheres had much higher microhardness at the waist and 45° location than the annealed hemisphere. The stress-relieved hemisphere exhibited the most variation in microhardness. The waist had the highest microhardness reading due to the highest amount of cold work. The pole was softer because it was not deformed as much during the spinforming operation. The direction of analysis (pole face versus radial face versus outward face) had little effect on the measured value. The annealed hemisphere was nearly uniformly soft.

Table 4 Vickers 500 g Microhardness of 21-6-9 Stainless Steel Hemispheres

Annealed plate prior to spinforming	test 1	201	test 2	197
		Pole face	Radial face	Outward face
As-spun sample A	waist	339.9	336.3	338.6
	45°	316.6	321.1	302.8
	pole	244.2	242.7	275
As-spun sample B	waist	322.2	331.6	334.4
	45°	307.8	334.6	306.4
	pole	240.3	250.9	236
Stress-relieved sample A	waist	341.9	342.4	311.5
	45°	277.2	292.7	273.8
	pole	254.3	253.5	231.7
Stress-relieved sample B	waist	327.3	309.5	325.8
	45°	292.1	326.2	285
	pole	186	190.8	187.7
Annealed sample A	waist	189.7	188	192.1
	45°	192.6	188.3	190.9
	pole	210.9	182.9	180.3
Annealed sample B	waist	222	247.8	225.5
	45°	187.3	190.5	188
	pole	180.1	189.1	198.1

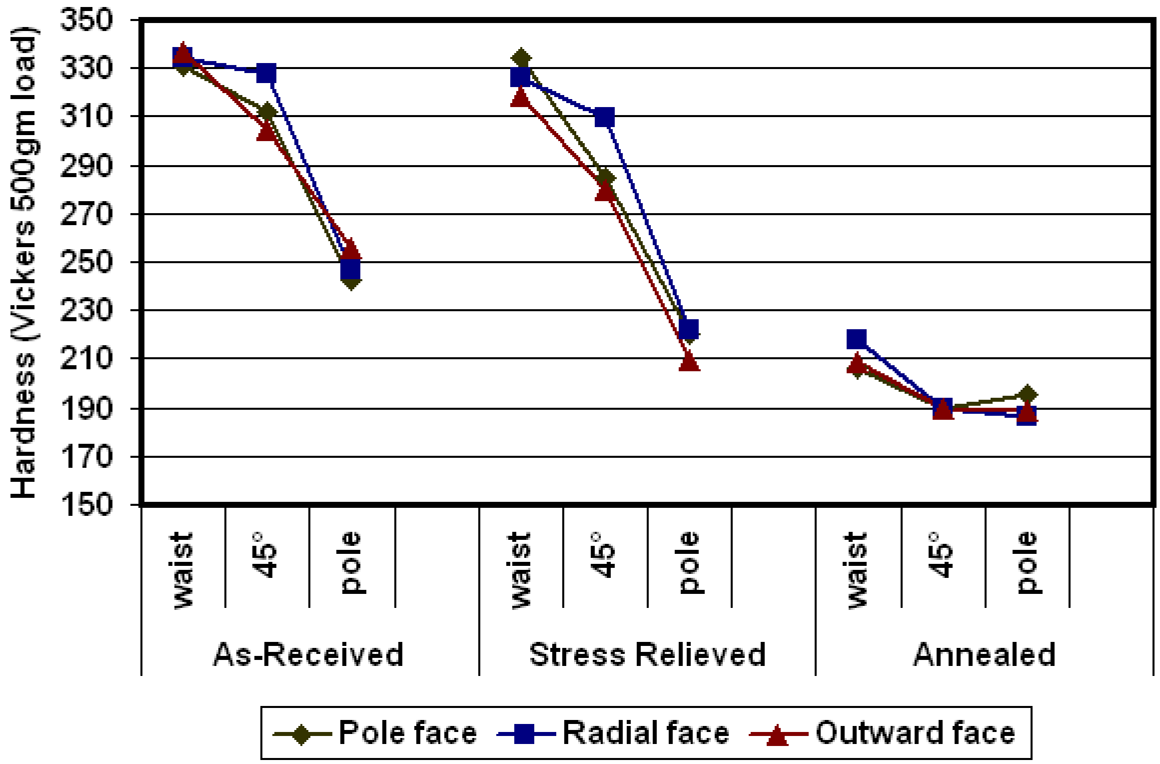


Figure 14 **500 gram load Vickers microhardness readings taken from metallographic samples for spinformed 21-6-9 hemispheres of each of the three conditions: as spun, stress-relieved at 1200° F for one hour then 1300° F for one hour, and annealed at 1900° F for one hour. Microhardness readings were taken from the metallographic samples from three locations on the hemispheres: near the waist, up about half way (45°), and near the top (pole). Readings were taken in three orientations: the surface normal to the pole, the surface normal to the radial direction, and the surface parallel with the outer surface of the spinformed part.**

Mechanical Properties

Miniature tensile bars were machined from the spinformed hemispheres in the waist, 45°, and pole locations and in the pole, radial, and circumferential directions. In Figure 7, the long, thin machined locations show from where the tensile bars were machined. Figure 15 is the dimensional sketch for the miniature tensile bar.

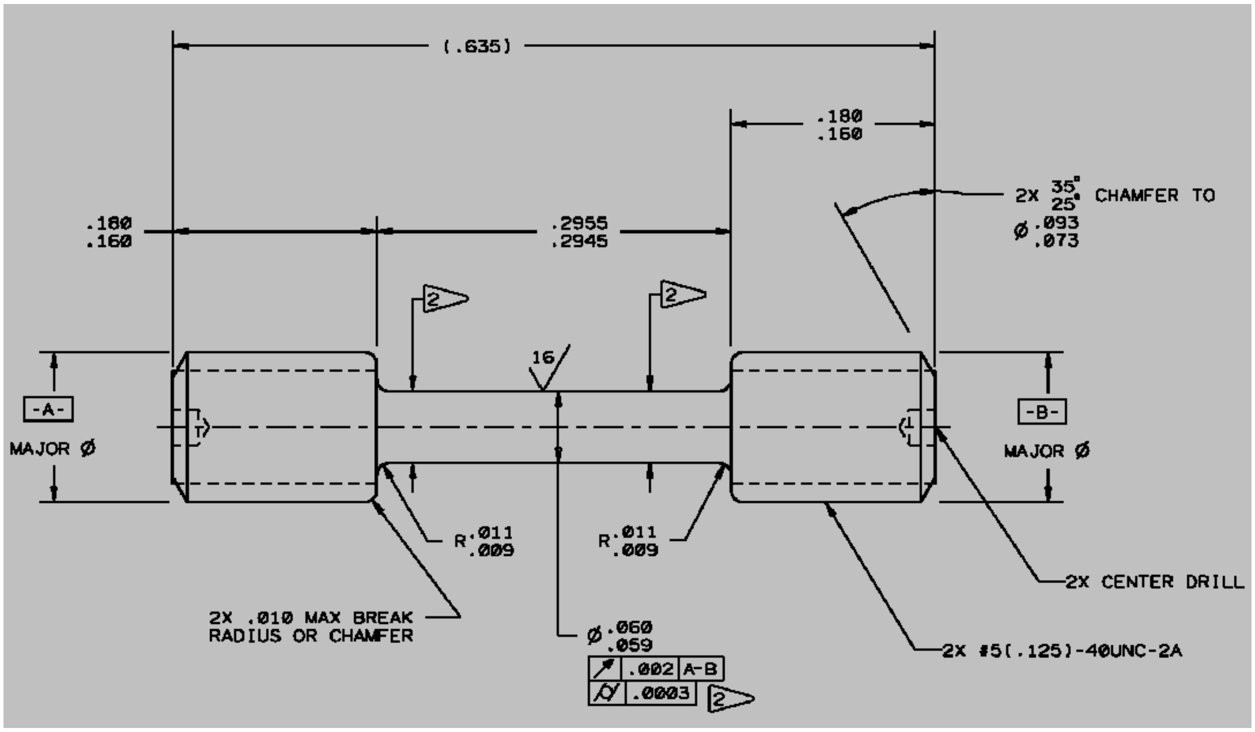


Figure 15 Miniature tensile bar dimensions per KCP drawing 1470302.

Table 5 presents the results of tensile testing for the hemispheres. Tensile bar direction—whether aligned with the pole or the circumference of the hemisphere—did not seem to have a large effect on strength values. Figure 16 shows the effect of each heat treatment on the yield strength and uniform elongation of the 21-6-9 stainless steel hemispheres. In general, the yield strength decreased due to stress relief and decreased more due to full anneal. The highest yield strength was a value of 136 ksi for the horizontal tensile bar in the waist location for the stress-relieved hemisphere. Annealing brought the strength into the range of 61-81 ksi.

Correspondingly, uniform elongation increased due to stress-relief and increased more due to full anneal. Uniform elongation was 27% or higher for all of the tensile tests for the hemispheres. This high ductility is typical for austenitic stainless steels due to the face-centered-cubic crystal structure.

Figure 17 plots the same tensile data versus sample location instead of heat treatment. Location on the spinformed hemisphere has an effect on tensile properties as well. Yield strength decreases from the waist to the 45° location to the pole for both the as-received and stress-relieved hemispheres. Consequently, elongation increases with the same progression from waist to pole. This trend occurs because the amount of cold work applied during spinforming is quite low near the pole, but steadily increases as the tool progresses toward the waist.

Table 5 Miniature Tensile Test Results for Spinformed 21-6-9 Hemispheres

Hemisphere Condition	Tensile Bar Location	Tensile Bar Direction	Yield Strength at 0.2% Offset (ksi)	Tensile Strength (ksi)	Elongation (%)	Reduction in Area (%)	Modulus of Elasticity (Mpsi)
As-spun	Waist	pole	118	141	44	77	27
As-spun	Waist	circumferential	128	150	30	77	27
As-spun	45º	pole	115	137	53	79	26
As-spun	45º	pole	115	137	46	80	29
As-spun	45º	circumferential	116	137	45	79	26
As-spun	45º	circumferential	120	138	45	81	28
As-spun	Pole	pole	92	125	64	82	30
As-spun	Pole	pole	83	122	68	83	25
As-spun	Pole	circumferential	72	118	71	83	28
Stress-relieved	Waist	pole	130	156	32	63	31
Stress-relieved	Waist	pole	136	169	27	67	25
Stress-relieved	Waist	circumferential	121	149	37	70	29
Stress-relieved	45º	pole	105	138	43	73	28
Stress-relieved	45º	pole	106	140	45	72	29
Stress-relieved	45º	circumferential	106	135	52	78	28
Stress-relieved	45º	circumferential	107	140	48	74	27
Stress-relieved	Pole	pole	77	120	68	80	27
Stress-relieved	Pole	pole	76	120	66	79	29
Stress-relieved	Pole	circumferential	82	121	58	84	27
Stress-relieved	Pole	circumferential	60	112	78	83	28
Annealed	Waist	pole	81	122	59	79	30
Annealed	Waist	pole	61	117	73	82	25
Annealed	Waist	circumferential	72	117	72	82	26
Annealed	Waist	circumferential	70	116	65	83	22
Annealed	45º	circumferential	74	126	58	76	28
Annealed	Pole	pole	61	116	77	80	24
Annealed	Pole	circumferential	65	115	69	83	27

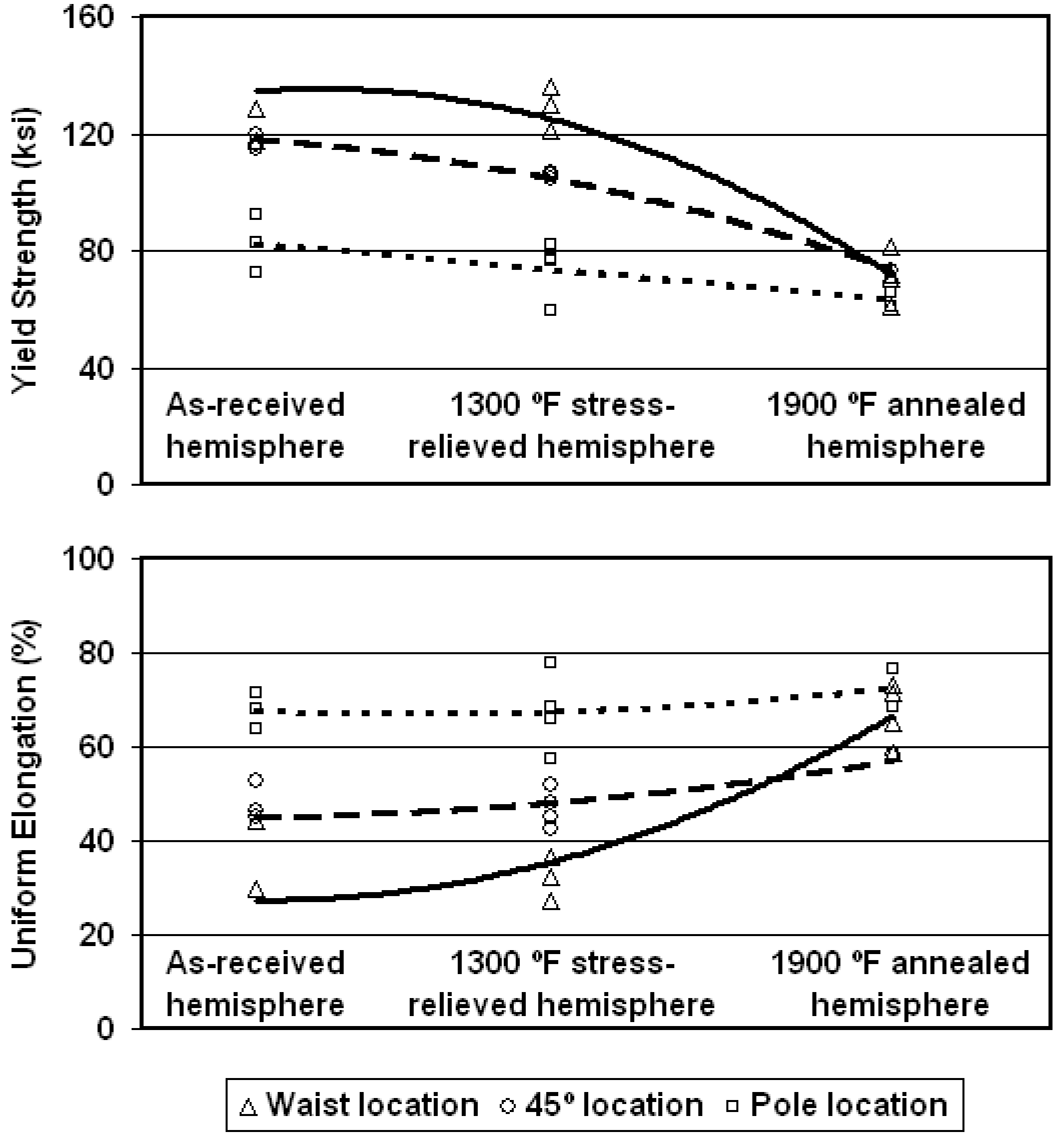


Figure 16 Effect of heat treatment on tensile yield strength and uniform elongation for 21-6-9 spinformed hemispheres. Observe that the waist location has the highest yield strength. The 45° location has slightly lower yield strength. The pole exhibits the lowest yield strength. The annealed hemisphere yielded within the range of 61-81 ksi regardless of location. As expected uniform elongation was generally lowest for the highest strength material and highest for the annealed hemisphere.

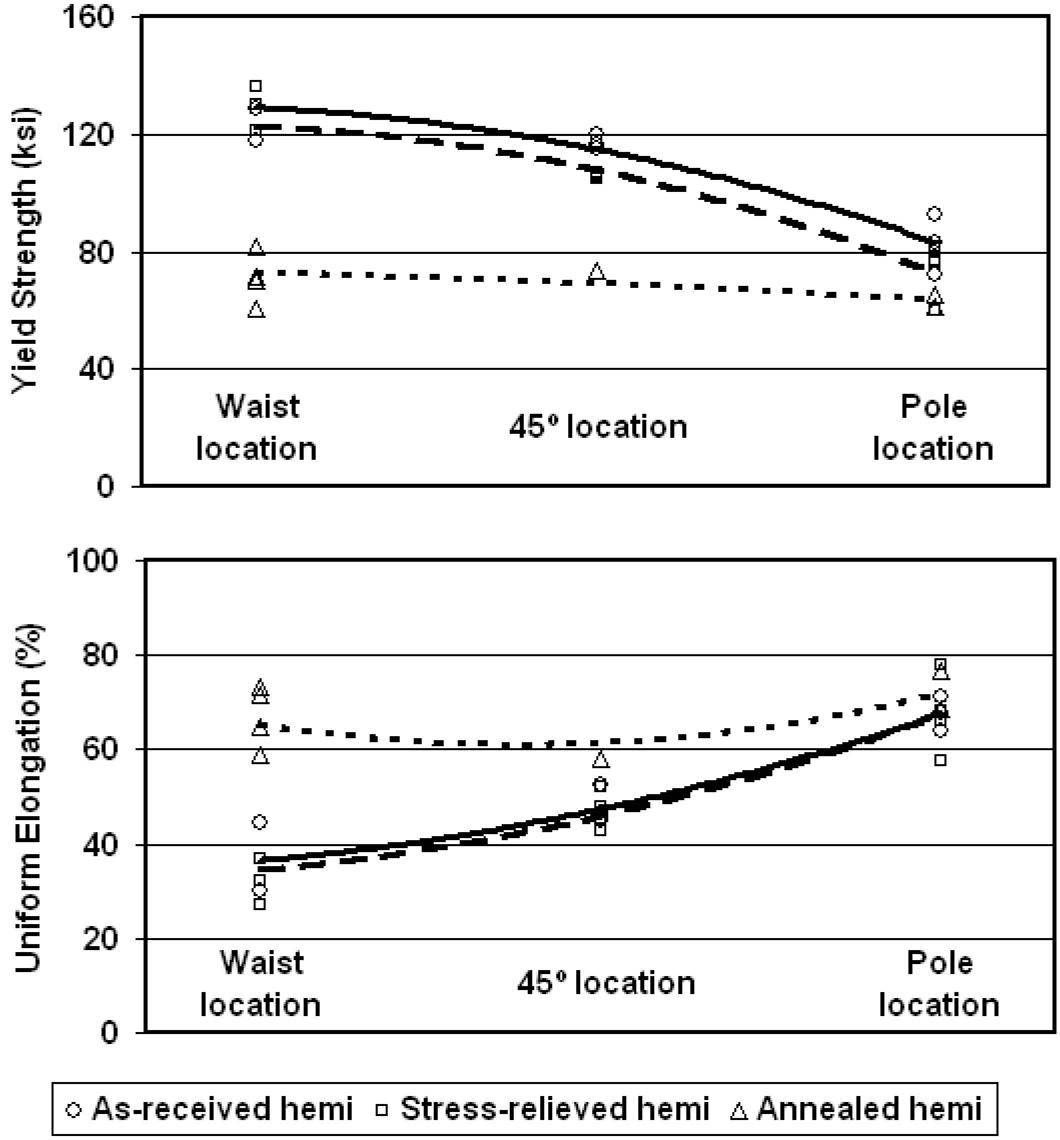


Figure 17 **Effect of specimen location on tensile yield strength and uniform elongation for 21-6-9 spinformed hemispheres. Yield strength decreases in the progression from the waist to the pole of the hemisphere for the as-received and stress-relieved hemispheres. Correspondingly, elongation increases along the same progression from waist to pole. The annealed hemisphere is relatively uniformly weak and ductile except near the pole where all of the hemispheres exhibit similar strength and ductility.**

Hardness Profiles

Figure 18 shows examples of remnants of the 21-6-9 spinformed hemispheres that were tested using a Rockwell scale. Rockwell B and C scales also were used as necessary, but all data was converted to the A scale for comparison. Due to the remnant size, the test setup was challenging, but care was taken to ensure that the work piece was stable and the readings were valid.

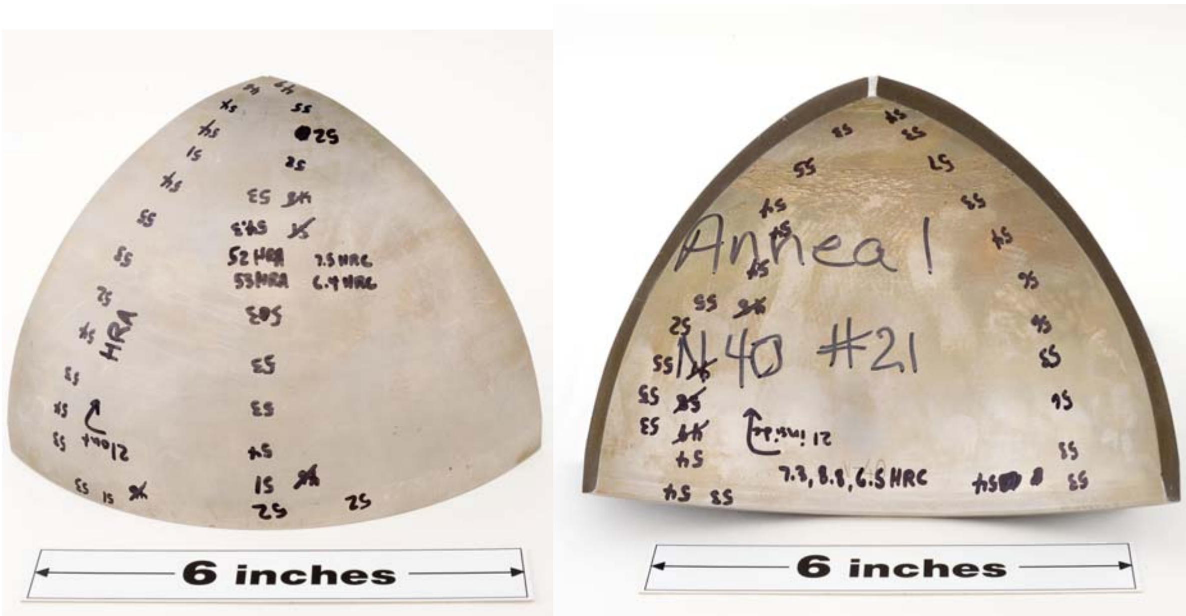


Figure 18 **Example photos showing the outside (left) and inside (right) hardness traces from the waist to the pole of the spinformed 21-6-9 hemispheres.**

Figure 19 presents the average hardness traces for each spinformed hemisphere from the waist to the pole. In general the outside hardness of the as-spun and stress-relieved hemispheres was fairly constant, but decreased toward the pole. However, the inside hardness for the as-spun and stress-relieved hemispheres spiked at the pole. This spike in hardness is due to the pressure and sliding that occurred on the surface of the material between the mandrel and tailstock. The hardness of the annealed hemisphere was relatively constant except near the pole. Since large grains were identified near the pole of the annealed hemisphere, it is possible that irregular grain growth and softening occurred due to over-annealing in that specific location.

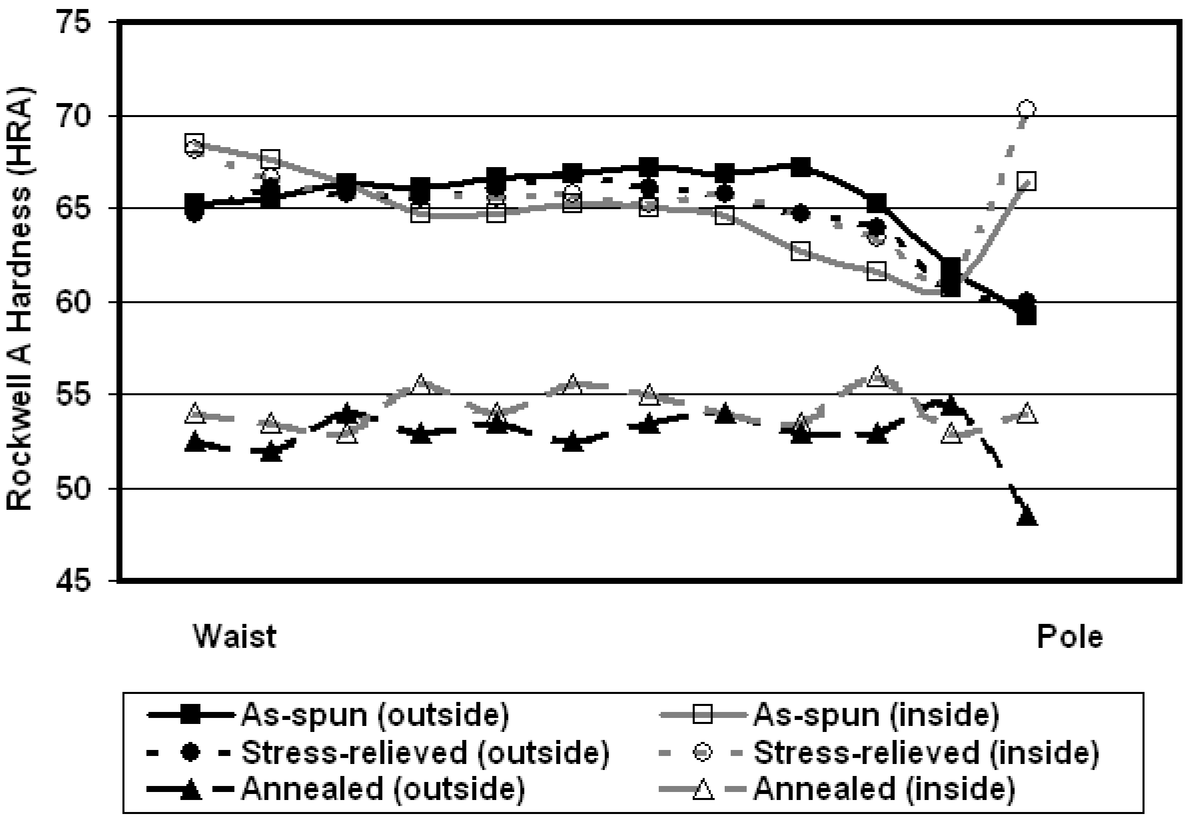


Figure 19 **Hardness traces from the waist to the pole of the spinformed 21-6-9 hemispheres of each of the three conditions: as spun, stress relieved at 1300 F for one hour, and annealed at 1900 F for one hour.**