Title: Improved Tubulars for Better Economics in Deep Gas Well Drilling using Microwave Technology

Annual Technical Progress Report

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1. Executive Summary

The objective of the research program has been to improve the rate-of-penetration in deep hostile environments by improving the life cycle and performance of coiled-tubing, an important component of a deep well drilling system for oil and gas exploration. The current process of the manufacture long tubular steel products consists of shaping the tube from flat strip, welding the seam and sections into lengths that can be miles long, and coiling onto reels. However, the welds, that are a weak point, now limit the performance of the coil tubing. This is not only from a toughness standpoint but also from a corrosion standpoint. By utilizing the latest developments in the sintering of materials with microwave energy and powder metal extrusion technology for the manufacture of seamless coiled tubing and other tubular products, these problems can be eliminated. The main objective of the project is therefore to develop a continuous microwave process to sinter continuously steel tubulars and butt-join them using microwave/induction process. The program started about four years ago and we have just completed Phase II.

In Phase I (which ended in February 2005) a feasibility study of the extrusion process of steel powder and continuously sinter the extruded tubing was conducted. The research program has been based on the development of microwave technology to process tubular specimens of powder metals, especially steels. The existing microwave systems at the Materials Research Laboratory (MRL) and Dennis Tool Company (DTC) were suitably modified to process tubular small specimens. The precursor powder metals were either extruded or cold isostatically pressed (CIP) to form tubular specimens. After conducting an extensive and systematic investigation of extrusion process for producing long tubes, it was determined that there were several difficulties in adopting extrusion process and it cannot be economically used for producing thousands of feet long green tubing. Therefore, in the Phase II the approach was modified to the microwave sintering combined with Cold Isostatic Press (CIP) and joining (by induction or microwave) the tubular parts into long coiled-tubing. Eventually, this process can be developed into a semi-continuous sintering process if the CIP can produce parts fast enough to match the microwave sintering rates. We have completed the sintering and joining of several tubular parts for conducting third party tests at Stress Engineering. This report summarizes the work completed in the pahse two and provides the test results performed by Stress Engineering.

The steel composition used in this work was 316L since the composition matching with the Quality tubing's QT-16Cr80 was not able in the maket in the pre-aaloyed powder form. The pre-mixed composition closely matching with QT-16Cr80 caused problems during sintering. Bonding experiments using 4 different braze powders were conducted and the process optimized to obtain high degree of bonding strength. For fabrication of green tubulars a large CIP unit was acquired and used for making upto 18 inch long green tubes. Microwave sintering experiments for continuous processing of the CIPed tubes completed and the samples were joined at Dennis Tool for further testing.

At Stress Engineering torsion, hardness, corrosion, crush, and fatigue tests were performed on 3/4" OD x 1/8" wall sintered stainless tubes.

Torsion Tests: Two brazed tubing assemblies (sintered stainless steel) were loaded in torsion to failure. The torque turn results from the two tests. Both samples started to yield at the same torque, however, the failure torque and rotation of sample T1 was much higher than sample T2. Both samples failed in the tube near the brazed joint. The results follow:

At yield: Samples T1 and T2 Torque at start of yield = 220 ft-lb Shear Stress = τ = 36,480 psi

At failure: Sample T1 Torque at failure = 393.8 ft-lb τ = 65,300 psi

Sample T2 Torque at failure = 273.3 ft-lb Shear Stress = τ = 45,320 psi

Crush Tests: Four 3” long samples were crushed radially (laying flat on their side) recording the load and displacement. The tubing was visually examined for cracking after being crushed. Samples C2, C3, and C4 cracked as they were loaded to 23.6 kips. Sample C1 was crushed flat at a load of 61.1 kips and it did not crack.

Micro-hardness Tests: Micro-hardness values were obtained on the OD of two samples. Values were obtained at six locations on either side and in the brazed joints. The samples were mounted, polished, prior to taking the readings.

Corrosion Tests: Corrosion tests were performed in an autoclave at 250°F and 500 psi for 96 hours with carbon dioxide bubbled through a 5% sodium chloride solution to provide 100% saturation of the brine. Test procedures from ASTM G31 and G111 were used as a guide.

Fatigue Tests: A fatigue test was performed on one sample using SES’s resonant fatigue machine. The resonant fatigue machine consists of two supports, a variable speed electric motor, drive housing, and dead weight housing. The variable speed motor rotates an eccentric mass in the drive housing clamped to one end of the sample and loads the pipe. The rpm of the motor is adjusted to load the sample near its natural frequency. The dead weight housing is clamped to the other end of the sample to balance the assembly. The sample failed in the turned down region of the sample after 22.16 million cycles at a stress range of 25.89 ksi. Figure 2 shows that the fatigue performance of the sample was comparable to girth welded 57mm OD Super Duplex A670 tubing.

The test results at the Stress Engineering can be summarized as the following:

- Some mechanical properties of the microwave processed tubes were better than the existing tubular, and some were just marginally better or a little inferior.
- The corrosion data in the Stress Eng. report suggests that the microwave sintered performed better than QT-900 and 13%Cr tubing material, but not as good as QT-16Cr.
- The crush results compare very well against previous microwave samples tested in the QT report from 2004, and performed quite satisfactorily. Some crushed samples became totally flat without showing any cracks.

Based on the test results at the completion of the Phase II, it is concluded that:

- Scale up and sintering of a thin wall common O.D. size tubing that is widely used in the market is still to be proved
- Further experimentation and refinement of the sintering process is needed to entice industry commitment, for example:
- Improved material characteristics would likely be required
- Improved consistency of material characteristics
- Actual manufacturing capability of microwave sintered, industrial quality, full length tubing will most likely require several million dollars of investment.

2. Abstract

The main objective of the entire research program has been to improve the rate-of-penetration in deep hostile environments by improving the life cycle and performance of coiled-tubing, an important component of a deep well drilling system for oil and gas exploration, by utilizing the latest developments in the microwave materials technology.

Based on the results of the Phase I and insurmountable difficulties faced in the extrusion and de-waxing processes, the approach of achieving the goals of the program was slightly changed in the Phase II in which an approach of microwave sintering combined with Cold Isostatic Press (CIP) and joining (by induction or microwave) has been adopted. This process can be developed into a semi-continuous sintering process if the CIP can produce parts fast enough to match the microwave sintering rates.

The main objective of the Phase II research program is to demonstrate the potential to economically manufacture microwave processed coiled tubing with improved performance for extended useful life under hostile coiled tubing drilling conditions. After the completion of the Phase II, it is concluded that scale up and sintering of a thin wall common O.D. size tubing that is widely used in the market is still to be proved and further experimentation and refinement of the sintering process is needed in Phase III. Actual manufacturing capability of microwave sintered, industrial quality, full length tubing will most likely require several million dollars of investment.

3. Experimental

The entire research program has been based on the development of microwave technology to process tubular specimens of powder metals, especially steels. In the reporting period the main focus was on selection of steel powder, preparation of green samples by CIP (cold isostatic pressing), microwave processing of test samples and bonding of sintered rods. The existing microwave sintering systems at Materials Research Lab and Dennis Tool Company were suitably modified to process the new steel powders. Also bonding experiments were conducted and preliminary strength data acquired on the bonded samples. The bonded samples were evaluated at the Stress Engineering for hardness, corrosion behavior, fatigue test, crush test and torsion test.

The experimental details of the microwave sintering, CIP and bonding had been already described in the last annual report. Here, we provide only the experimental details of the mechanical tests performed at the third party (Stress Engineering).

Several 12” long test samples were microwave sintered at Penn State University using the pre-alloyed 316L material and optimized sintering conditions in the regular vertical continuous 2.45 GHz, multimode sintering system. This system was suitable modified to process such tubular samples. The green tubes of 12 inch in length and nominally 1.15” OD 1” ID were prepared using cold isostatic pressing (CIP). The binder burn out of these green samples was conducted in a conventional furnace separately. Before attempting 12 inch tubes, several smaller tubes (3-5 inch) were also fabricated for crush and corrosion tests. All sintered samples were sent to Dennis Tool Company (DTC) for machining and brazing to make long (up to 48 inch) tubes for fatigue tests. At DTC these samples were cut, machined, brazed, as needed and finally were packaged for testing at Stress Engineering Services. The experimental procedure for these tests is described below:

Torsion Test: Two brazed tubing assemblies (sintered stainless steel) were be loaded in torsion to failure. The tests were performed in a machine that includes an encoder to monitor the rotation and a torsion load cell to monitor the torque. Figure 3.1 shows a sample in the machines both with and without the safety shield. The machine is normally used to compare the friction characteristics of thread lubricants, but it is well suited for the torsion failure tests. Both samples were loaded to failure. The torque and rotation was recorded by a high speed data acquisition system. The yield and failure torques were used to calculate the yield and failure shear stresses based on the nominal pipe OD and ID. Since the torque machine is set up to use standard sockets, the shanks of ¾” bolts were turned down to slid into the ends of the samples and welded to the tube, resulting in hex bolt head at each end of the samples, as shown in Figure 3.2.

Crush Test: Four 3” long samples were crushed. A 20,000 lb capacity Baldwin load frame was used to crush the tubing. The displacement and load required to crush the tubing radially (laying flat on its side) was recorded. The tubing was visually examined for cracking after being crushed.

Micro hardness Test: Two tubing samples containing brazed joints were supplied for micro-hardness evaluation of the brazed joints. A view of the two brazed joints (following sectioning) is presented in Figure 3.3. As can be seen, both tubing samples were sectioned along their longitudinal axes. Half of each brazed sample was subsequently mounted in plastic for examination of the structure and hardness of the brazed joint. The mounted portions of the tubing samples are shown toward the top of the field of view in Figure 3.3A.

Corrosion Test: Corrosion tests were performed in an autoclave at 250°F and 500 psi for 96 hours with carbon dioxide bubbled through a 5% sodium chloride solution to provide 100% saturation of the brine. Test procedures from ASTM G31 and G111 were used as a guide.

Fatigue Test: Stress Engineering Service’s resonant fatigue test machine was used for the fatigue tests. The resonant fatigue machine consists of two supports, a variable speed electric motor, drive housing, and dead end housing. Figure 3.4 shows the sample in the fatigue machine. The variable speed motor rotates an eccentric mass in the drive housing clamped to one end of the sample and loads the pipe. The rpm of the motor is adjusted to load the sample near its natural frequency. The applied load produces the same a sinusoidal alternating stress at every point around the circumference of the sample. The dead weight housing is clamped to the other end of the sample to balance the assembly. The test sample length required and the resulting test frequency were determined using a finite element model. Axial strain gages on the outer diameter of the center section of the tube were used to monitor the bending strains during the fatigue test. Both bending strains and number of cycles were monitored and recorded by the fatigue data acquisition system. Each minute the data acquisition software recorded the average maximum and minimum strains for each strain gage along with the number of cycles.

Two mechanisms are normally used to stop the test if the sample has cracked through the wall during fatigue testing: wet detectors and pressure switches. However, the test was performed without water in the sample the test stopped when the crack was large enough to reduce the stiffness of the sample enough to lower the natural frequency of the sample so that the stress range will drop.

Test Procedure The general steps, involved in testing the sample, were:

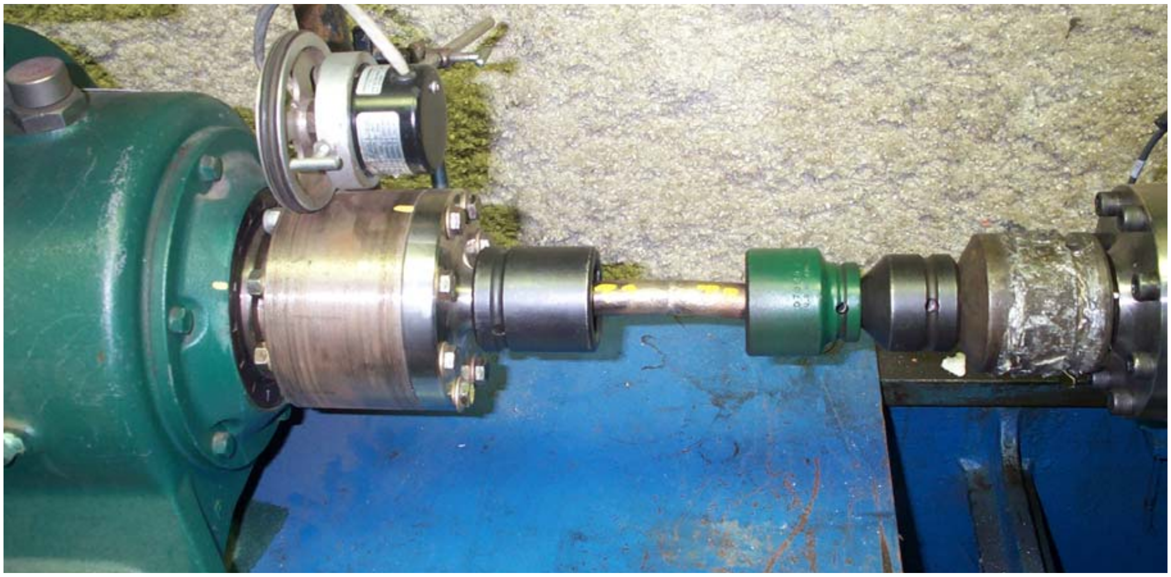
1. Measure and record the wall and outer diameter at each strain gage location.
2. Install the strain gages to monitor the fatigue strains.
3. Load the sample in the fatigue test machine.
4. Adjust the rpm of the test machine to achieve the desired strain range.
5. Log strain amplitude, frequency, and cycles.
6. Fatigue test the sample until failure.
7. Inspect the sample.
8. Document the test including fatigue test strains and cycles.

Test Sample

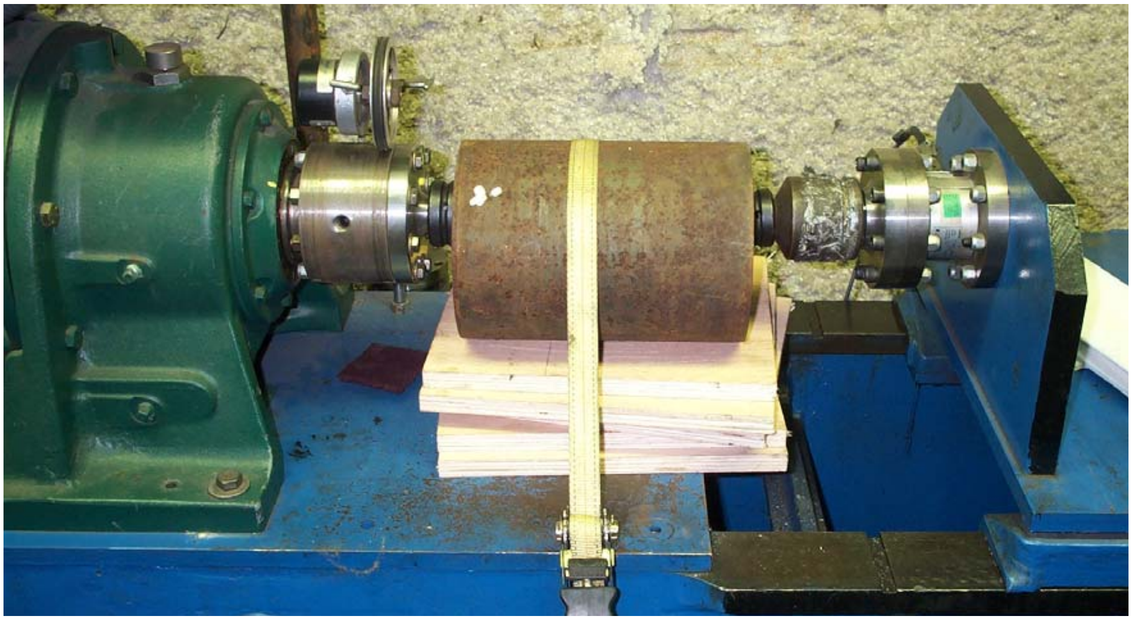
In order to get a sample long enough to test in the resonant test machine five short (about 9" long) sections of tube were joined with stainless steel sleeves and brazed together, as shown in Figure 3.5. The center of the middle section was turned down so the failure would occur in the middle of the sample. A total of eight axial strain gages were installed to monitor the test, four at 90º intervals around the circumference on either side of center of the turned section in the center of the sample. The sets of four gages were offset by 45º so that there was a gage every 45º around the circumference of the sample as shown in Figure 3.5. The ID and OD at the strain gages were not concentric. The OD at the gages was 0.799" and the ID was 0.595". After the sample broke, the diameters were measured along with the wall thickness at each strain gage. The thicknesses at the gages are given below.

Pipe Wall Thickness at Each Strain Gage

Gage Number	Wall Thickness in.
1	0.103
2	0.128
3	0.131
4	0.126
5	0.109
6	0.084
7	0.074
8	0.077



A. Sample In Torque Machine (without safety shield)

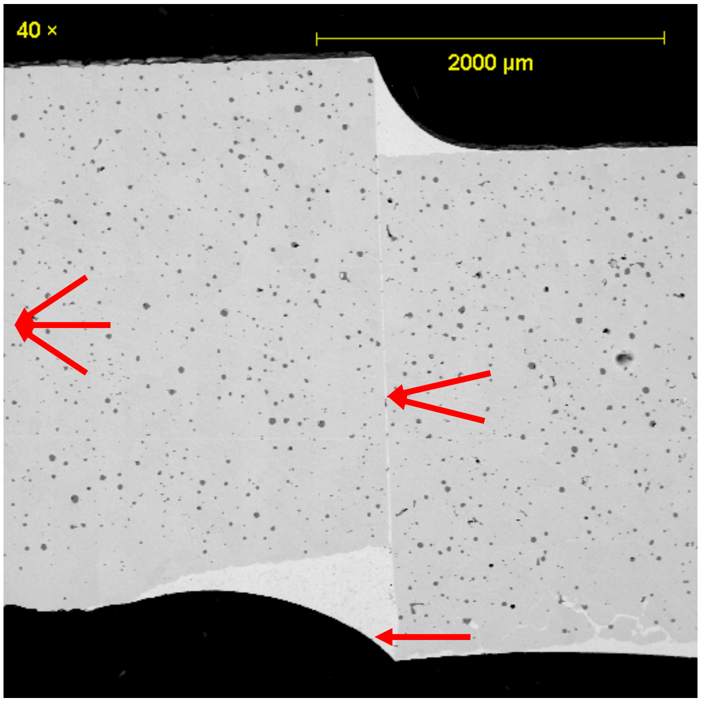


B. Sample In Torque Machine (with safety shield)

Figure 3.1: Torque test performed on the sample: Samples in torque machine: A. without safety shield, and B. with safety shield.

Figure 3.2: Torsion test samples before test.

A. A view of the samples containing the brazed joints (taken following sectioning and mounting).



B. A back-scattered electron image of a cross-section through the joint in sample 1. Locations where hardness measurements were made are identified.

Figure 3.3: Microhardness photographs

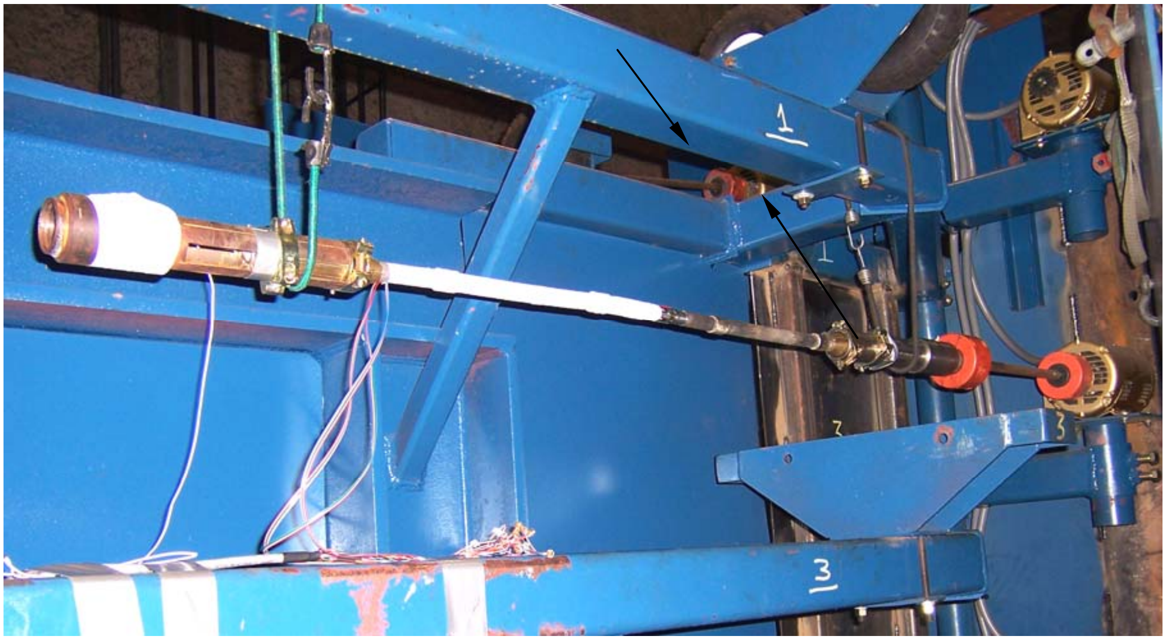
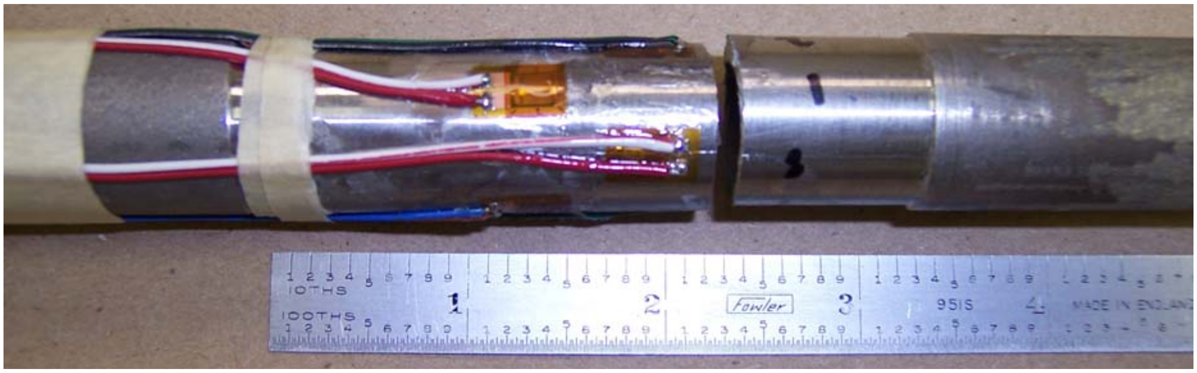


Figure 3.4: Sample in fatigue test machine.

Overall Sample

Center Section with Typical Brazed Sleeves Connecting Sections of Sample



Strain Gages in Center of Turned Section

Figure 3.5: Sample in the fatigue machine after testing at different magnifications.

4. Results and Discussion

Torsion Test:

Figures 4.1, 4.2 and 4.3 show the samples after the tests. Black axial lines were drawn the samples prior to the tests. Figure 4.1 shows that the lines on sample T1 are helical while the lines on sample T2 are almost straight. Sample T1 failed 0.15" from the braze joint while the failure in sample T2 was 0.25" from the braze joint. The ends of the two failed samples are shown in Figures 2.4 and 2.5. The torque rotation plots, for the two samples, are shown in Figure 4.4. Sample T1 twisted 132.4 degrees prior to failure while sample T2 only twisted 24.6 degrees. Both samples started to yield at 220 ft-lb. The failure torques were 393.8 ft-lb for sample T1 and 273.3 ft-lb for sample T2.

Shear stresses at the yield and failure torques are calculated based on the tests of samples T1 and T2 using tube dimensions of 0.79" OD and 0.56" ID. The shear stresses at yield and failure were:

At yield: Samples T1 and T2

Torque at start of yield = 220 ft-lb

Shear Stress = τ = 36,480 psi

At failure: Sample T1

Torque at failure = 393.8 ft-lb

Shear Stress = τ = 65,300 psi

Sample T2

Torque at failure = 273.3 ft-lb

Shear Stress = τ = 45,320 psi

Isolating the braze joint in two samples for torsion (45,320-65,380 psi) was intended to provide feedback concerning a proposed joining method for future microwave tubing samples to be fabricated in this manner. The results indicate that the shear failure stress of the microwave sintered material and the corresponding braze joint in one case is above nominal and in one case below nominal for 316L type stainless steel (annealed). The typical tensile stress shown for 316L stainless is 78,000 psi (annealed) and the corresponding shear stress (@ .75 x 78,000) is 58,500 psi. The Quality Tubing materials for coiled tubes is QT16Cr for example, (taken from QT report – 2004') shows an average tensile strength of 122,470 psi.

Crush Test:

Prior to the test the samples were numbered C1, C2, C3 and C4, as shown in Figure 4.5. Figure 4.6 shows a sample in the load frame. Figure 4.7 shows the load displacement plots for the four samples. The load frame has low and high low ranges and the initial tests were performed by loading the samples to the maximum of the low load range. Samples C2, C3, And C4 cracked during loading at the low load range. Sample C1 did not crack at the low load range and it was subsequently loaded until it was flat. Sample C1 did not crack.

The supplied microwave samples for the crush tests were not the best we have seen (23.6-61.1 kips). In crush tests performed several times at Dennis Tool Company, it was noted that the sintered density of the sample plays a critical role in its resistance to cracking during the test. With good density, we have seen consistent results that are similar to the "C1" crush test sample above. These results compare very well against previous microwave samples tested in the QT report from 2004, and performed quite satisfactorily. Some crushed samples became totally flat without showing any cracks as shown in Figures 4.8 and 4.9.

Micro hardness Test:

Following polishing and etching, micro-hardness measurements were made on the brazed joints in the mounted samples. The original Vickers micro-hardness measurements were converted to equivalent Rockwell B values. For both samples, hardness reading # 1 was taken in the braze alloy, near the surface of the sample, outside of the thin braze zone. Reading # 2, on the other hand, was taken near mid wall in the center of the thin braze alloy layer. Reading # 3 for each sample was taken in the wall of the tube, remote from the braze zone. The approximate locations of the hardness measurements are shown by the arrows Figure 3.3 B and Figure 4.10 A.

The hardness of the braze alloy ranged from approximately Rockwell B 70 to Rockwell B 79. The hardness of the tube wall, on the other hand, was somewhat higher, in the range of Rockwell B 81 to Rockwell B 92.

The structures of the braze joints can be seen in Figure 3.3 B and Figure 4.10A. The lighter areas in these photographs thus represent regions of higher average atomic weight and the dark areas are from regions of relatively low average atomic weight. The braze alloy can thus be seen to have a somewhat higher average atomic weight that the wall of the adjacent tubes. The tubes, on the other hand, can be seen to contain a relatively high concentration of low average atomic weight particles or precipitates. A high magnification view (originally taken at 500 X) of the braze joint in sample # 1 is shown in 4.10B, from which we estimated the maximum thickness of the braze alloy in the joint to be approximately 0.0004 inches (0.4 mils). The compositions of the braze alloys and the tube walls were subsequently evaluated using the “energy dispersive x-ray spectrometer”, (EDS), attachment of the SEM. An SEM-EDS scan gives a semi-quantitative estimate of the chemical elements that are in the small area of the sample surface that is excited to emit x-rays by the focused electron beam of the SEM. This study shows the compositions of the tube base metals, and it was found that the metallic part of the tubes consisted primarily of iron, chromium, nickel, manganese and molybdenum. A review of alloy literature showed that the tube wall apparently came closer to satisfying the chemical requirements for Nitronic 60 than those for Type 316 S.S., as initially suspected. The braze metal, on the other hand, was found to consist primarily of copper, manganese, nickel and iron. The tube walls also contained a relatively high concentration of low average atomic weight particles. These particles were found to consist primarily of oxygen, silicon and manganese.

Corrosion Test:

Two samples of tubing were sent to an outside test facility (Honeywell International, Houston, Texas) for corrosion testing. The two samples (after their return from the corrosion testing) are shown in Figure 4.11. The samples were exposed for a period of 96 hours to 5% NaCl brine at room temperature, under 500 psi carbon dioxide. The measured corrosion rates for the two samples were found to be 4 mils/yr and 14 mil/yr. This compares with QT-16Cr rates of 2-3 mils/yr., QT-900 rates of 19-21 mils/yr., and 13% Cr rates of 20-28 mils/yr. (data via Quality Tubing website).

The corrosion data suggests that the microwave sintered performed better than QT-900 and 13%Cr tubing material, but not as good as QT-16Cr. Throughout our experiences in sintering with the microwave system, we have consistently seen noticeable, and sometimes, dramatic improvement in corrosion resistance. The samples supplied to Stress Engineering were not surface prepped in any way (these samples were EDM cut at each end) and perhaps would have given much better results if ground finish on all surfaces before testing.

Fatigue Test:

The test was performed at three different stress ranges. The first two stress ranges were due to changes in the response of the sample during the test and the third was an intentional increase in the stress range to shorten the test. The three stress ranges were combined into one equivalent stress range using Miners rule and a stress range to cycle relationship with m= -4. Table 4.1 gives the strain gage results for the three stress ranges and the associated cycle counts. The equivalent stress range and cycle count are given in Table 4.2. Figure 4.13 compares the sintered tube test results to the BS 7608 B curve from British Standard "Fatigue design and assessment of steel structures, 1993". The results are also compared to the mean results for 57mm OD Super Duplex X670 umbilical tubes. The Super Duplex results were taken from "Effect of Reeling on Welded Umbilical Tubing Fatigue" OMAE 2006-92579. The fatigue failure surfaces are shown in Figures 3.5 and 4.12. The sample failed near the center of the turned down section in the middle of the sample.

The test data seems to indicate that in some respects, the microwave sintered tubing performed as well as the comparable super duplex stainless steel tubing. Most notably is the fatigue test in which the failure of the microwave sample fell directly in line with the duplex failure slope. The microwave fatigue sample was made up of 5 separate pieces of tubing which were brazed together with a high strength, high temperature braze alloy. To ensure the test would focus on the sintered material and not a braze joint (which has been isolated and tested for strength), each braze joint was reinforced with a small, machined outer sleeve (Figure 3.5). The center section (mid-point along the length of the tubing assembly) O.D. was machined down to produce a clean surface on the O.D. Here, strain gauges were mounted allowing accurate measurements throughout the test. The wall thickness in this area of load concentration was somewhat variable due to the machining and less than perfect concentricity of the sintered O.D. to I.D.

The commonly used fatigue test in the coil tubing industry is a low cycle, high deformation test using a pressurized section of tubing (minimum sample of 72" in length). Due to processing constraints, a homogeneous, uniform 72" microwave sintered sample could not be provided without braze or weld joints along the sample. In order to isolate the microwave material characteristics and eliminate a joining method testing variable, a high cycle, low deformation test was used at Stress Eng. This constrained the testing to a uniform, homogenous area along the microwave tubing sample.

Figure 4.1: Torsion test sample after test (T1 top, T2 bottom)

Figure 4.2: Sample T1 after test.

Figure 4.3: Sample T2 after test

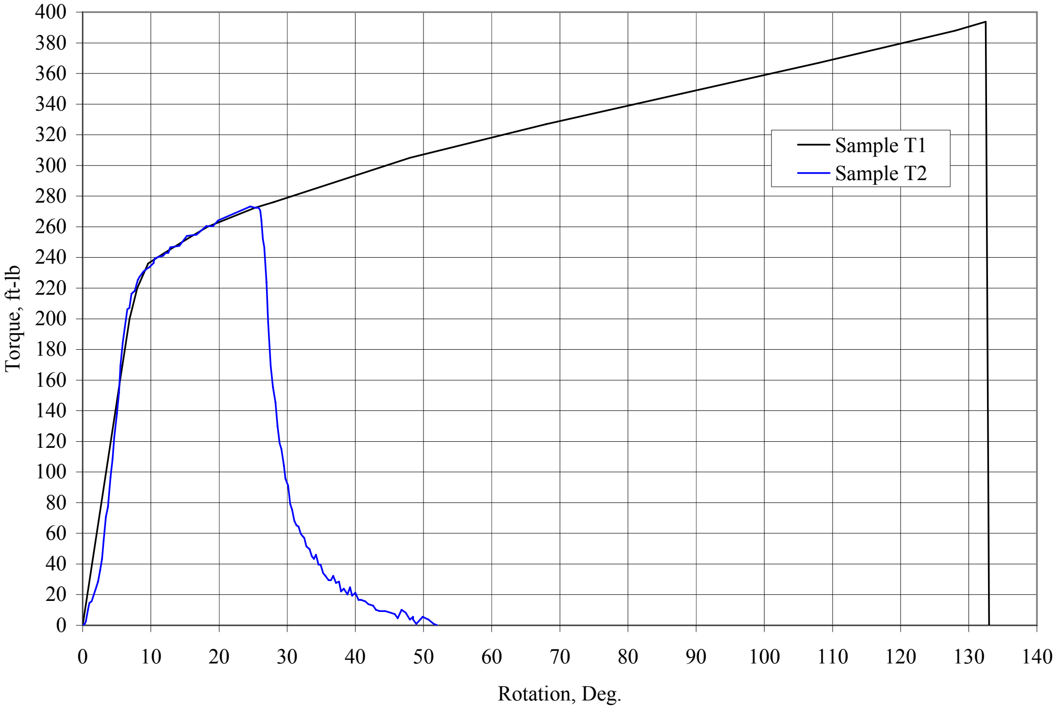


Figure 4.4: Failure torque test results.

Figure 4.5: Compression test samples.

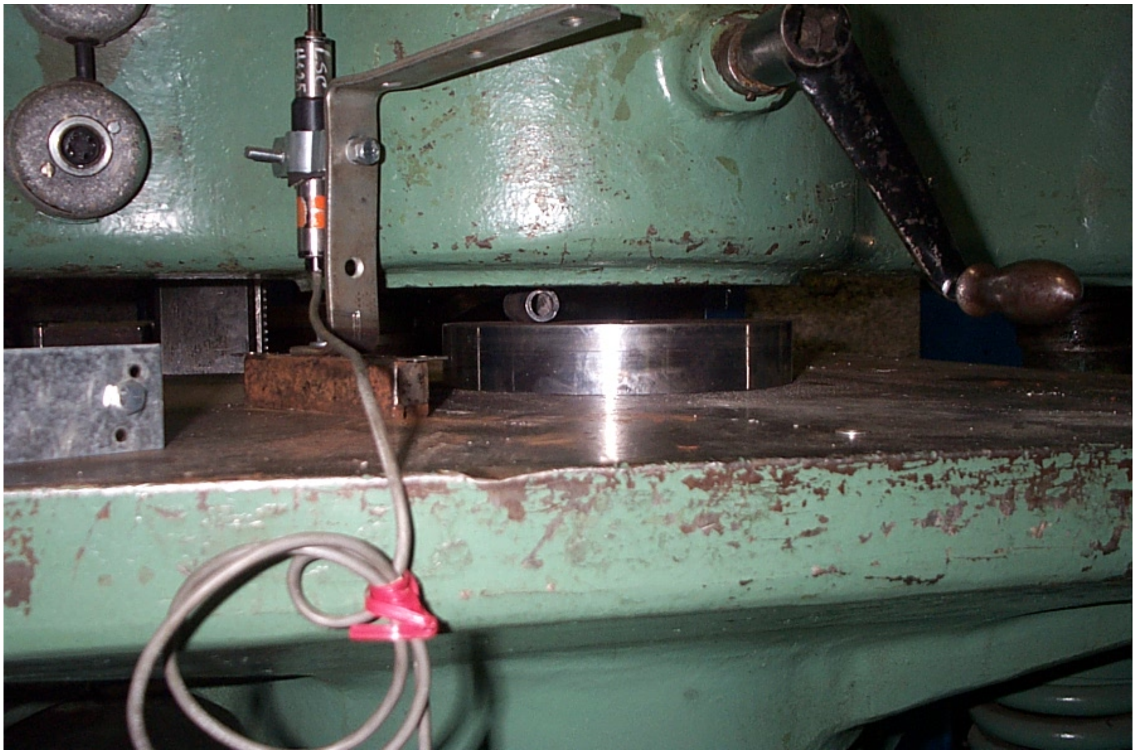


Figure 4.6: Compression sample in load frame.

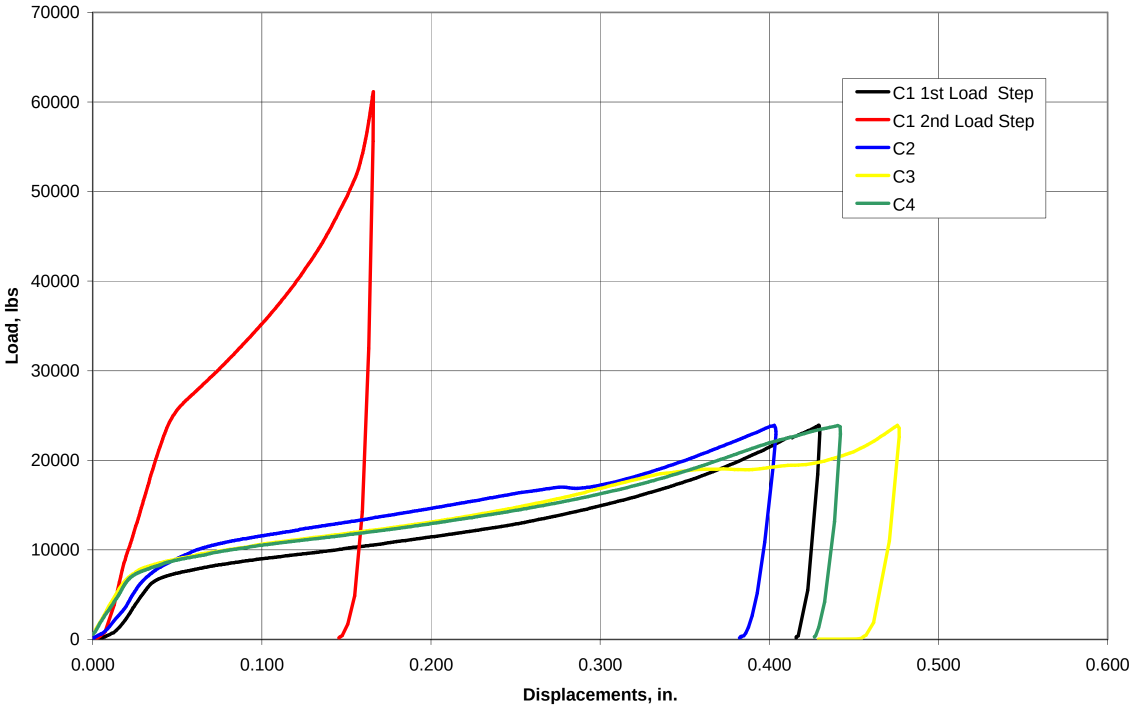
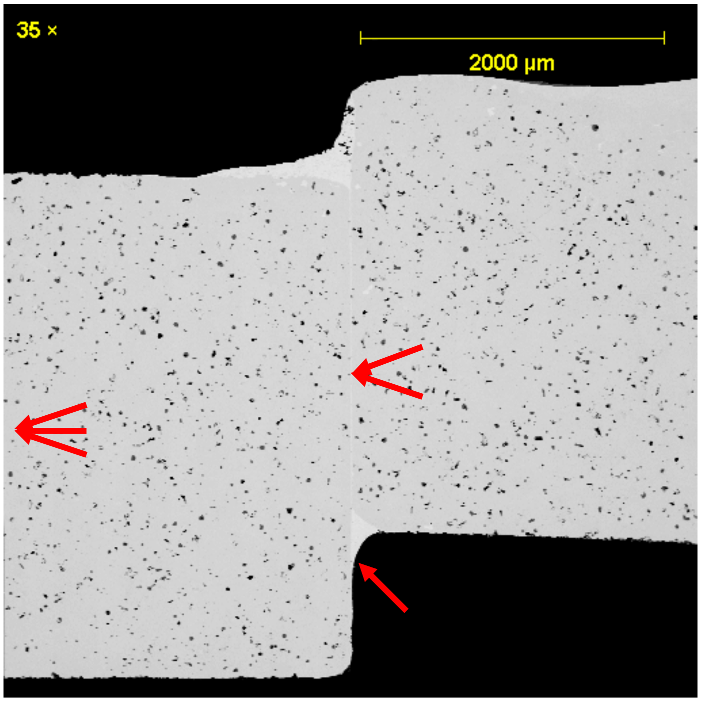


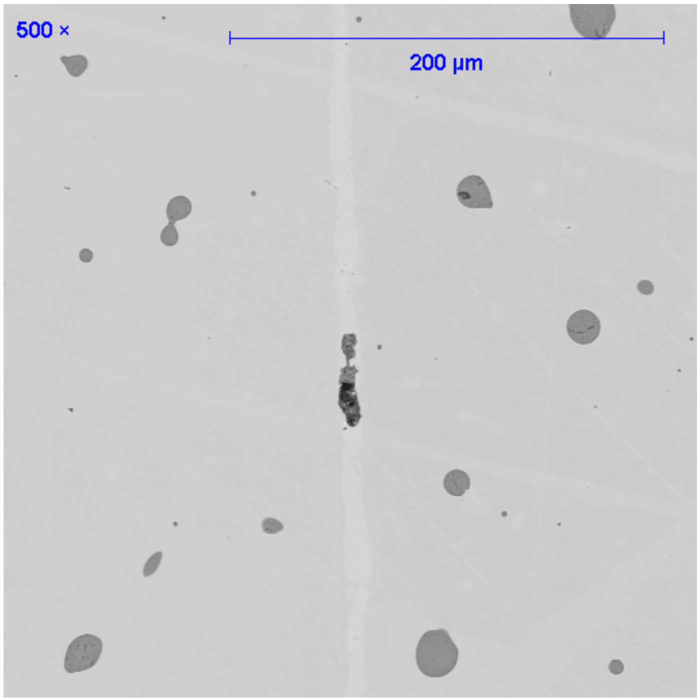
Figure 4.7: Crush test load displacement curves.

Figure 4.8: Crush test samples after the test,
Sample C1 (left) did not crack. Samples C2, C3, and C4 (right) cracked.

Figure 4.9: Crush test samples after the test,
Sample C1 (left) did not crack. Samples C2, C3, and C4 (right) cracked.



A. **A cross-section through the joint in sample 2.**



B. **A close up view of the braze joint in sample 1.**

Figure 4.10: Microhardness photographs.

Figure 4.11: Samples used for corrosion tests.

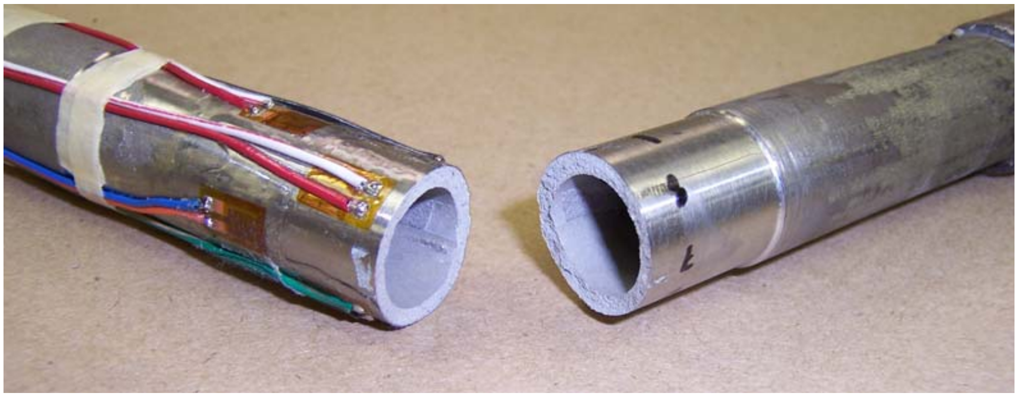


Figure 4.12: Fatigue failure surfaces (0.9” from right edge of turned down region)

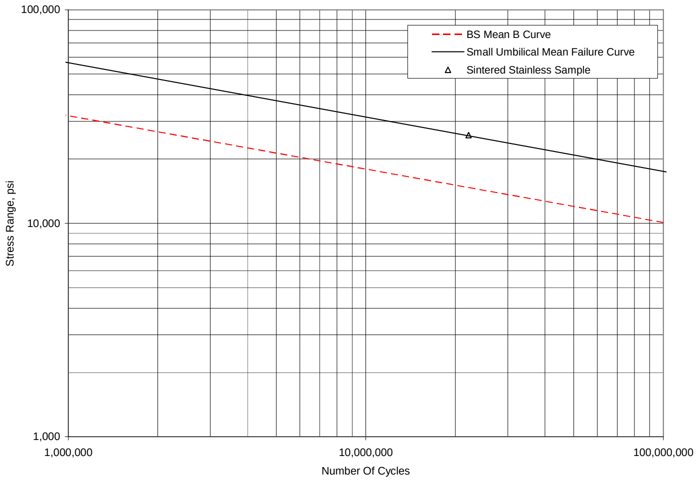


Figure 4.13: Sintered stainless tube fatigue results.

TABLE 4.1
Fatigue test strain ranges

Sample		Strain Range , ue										Max.Stress Range, psi	Cycles
		SG 1	SG 2	SG 3	SG 4	SG 5	SG 6	SG 7	SG 8	Average	Maximum		
1	1	308	335	340	292	297	378	399	347	337	399	11,983	965,121
	2	740	728	765	747	718	793	863	860	777	863	25,902	9,638,313
	3	-	-	-	-	-	1554	-	1567	1560	1567	47,003	1,151,242

TABLE 4.2
Equivalent fatigue results

N = A (S^-m) Basic form of the S-N curve
S = stress range, mpa
m = -4 slope of "B'" curve
n = 1 number of failures
A "B" desig 1.01E+15 for the "B" Design Curve used to calculate Damage
SCF = 1 Equivalent Connector Stress Concentration Factor (adjust to A survival above A design)

	1st Test			2nd Test			3rd Test			Total	Equivalent	Equivalent	Equivalent
Sample	Pipe Stress Range, psi	Cycles	Damage	Pipe Stress Range, psi	Cycles	Damage	Pipe Stress Range, psi	Cycles	Damage	Damage	Stress Range mpa	Stress Range psi	Test Duration Cycles
1	11983	965,121	0.0445	25902	9,638,313	9.7047	47003	1,151,242	12.5705	22.3197	178.58	25893.68	22,166,982

5. CONCLUSIONS AND RECOMMENDATIONS

- It is possible to sinter in microwave continuously steel tubular products. However, since the sintering temperature window is quite narrow, the scale-up process therefore has to be developed very carefully.
- Cost per foot to process Microwave Tubing would be difficult to estimate at this time due to lack of enough available processing data and a prototype microwave sintering system.
- Some mechanical properties of the microwave processed tubes were better than the existing tubular, and some were just marginally better or a little inferior.
- Scale up and sintering of a thin wall common O.D. size tubing that is widely used in the market is still to be proved
- Further experimentation and refinement of the sintering process is needed to entice industry commitment, for example:
- Improved material characteristics would likely be required
- Improved consistency of material characteristics
- Actual manufacturing capability of microwave sintered, industrial quality, full length tubing will most likely require several million dollars of investment.