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

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## 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 project is therefore to develop a continuous microwave process to sinter continuously formed/extruded steel powders. In order to begin the program quickly, three paths of investigation were started. They were metal powder composition development, process/equipment development, and extrusion development. This is a summary of Phase I of the three-phase program.

1) Since of the several steel compositions that were typically used for making coil tubing none were available in pre-alloyed powder form for sintering, the powder compositions were obtained by mechanically mixing the elemental powders. The samples of these compositions after microwave could not achieve full densification. Since this steel composition was not reported to have sintered via powder metal methods, there was no data available on its sintering conditions. So a systematic study to identify the optimum sintering conditions was conducted.

In the course of the investigation, it was found that sintering atmosphere is critical for obtaining good sintering. The best results were obtained with pure or 75% hydrogen atmosphere. Usually in PM sintering processes pre-alloyed mixes are used for consolidation since this provides higher densification, more uniform sintering, and better properties. Since the pre-alloyed starting powder of typical compositions for coiled tubing were not available in small quantities and high chrome steel was being investigated already, four chrome steel compositions were selected that are known to exhibit high strength and high corrosion resistance characteristics.

The four compositions were sintered and samples were characterized. Of these, 316L was found to be the best and was selected as the final composition for demonstration microwave experiments. The results showed that nearly full density at $1340^{o}$C/10 min was achieved. When the microstructural data was examined, everything was quite consistent with the density results and showed very little porosity. This was just like the microstructure observed in conventional coil tubing steels. This composition was therefore chosen for the next phase.

2) The process/equipment capabilities were investigated by taking standard powder metal steel tubes and solid bars wich were sintered in the existing microwave systems. After carefully fine-tuning and optimization of the processing conditions, excellent sintered samples of both tubular and solid cylindrical shapes were obtained.

In order to test the continuous movement of samples during sintering, several cylindrical specimens were prepared and stacked in order to move many powder metal samples at the same time. The processing conditions were adjusted to obtain the optimum sintering temperature using alumina crucibles to separate the samples. The samples sintered well and the shapes were good.

3) For demonstration of the sintering of long tubular specimens and for expediency, both extrusion and cold isostatic pressing (CIP) methods were tried. First, CIP was used to make 12 inch long solid and hollow tubes of nominally 1-1.5 inch diameter. The modified microwave system was used for

continuous sintering of 12-inch CIP tube. After process optimization, good microstructures were obtained. The movement rates of up to 10-min/inch and temperature of up to 1305$^{o}$C were needed.

In order to improve sinter-ability with liquid phase sintering and the mechanical properties, a preliminary study of dopants in steels was made using conventional methods initially. For this purpose a few wt % of YAG (Yttrium aluminum garnet, $Y_3Al_5O_{12}$) or $Y_2O_3$ were added to 316L. The results were that an optimum value of 5% of YAG that enhances the strength by over 50 % over the pure 316L. As in other materials, further improvement to the strength and other properties can be obtained by microwave sintering. These values are very compatible with the strength values normally required for coiled tubing applications. Similarly, the hardness values have also substantially increased.

4) For extrusion of powders, experiments of the sintering of the steel powders and binders in pellet forms demonstrated that nearly full density has been obtained. The sintering experiments have shown that iron materials are difficult to sinter to full density. The parts tend to have more porosity at the surface as compared to the center. This appears to be due to the initial poor coupling of the microwave energy to the iron powders as the tests for bonding two discs of iron powder (bonding of lengths of pipe together) were not successful until a cobalt cemented carbide mix was tested. The carbide coupled well and heated the iron discs to melting. When nickel and cobalt powders were tested in contact with the iron powder, they sintered well but the iron powders did not, and bonding was minimal. Also, 5% mixtures of the various powders in iron sintered well. In order to demonstrate the sinter-ability and extrude-ability of steel in a timely fashion, a program with typical organic binders was completed and demonstrated that extrusion of powder metal rods was successful. However, long de-binding times will be necessary with this system.

The extrusion of steel into tubes will require development of unique techniques for extruding tubes that are transferred to the microwave applicator in a manner consistent with the sintering times that will be needed. The development of a de-binding method that can be matched with the extrusion times and sintering times is a challenge. The maintaining of the proper de-binding/sintering atmosphere both inside and outside of the green and sintered tube will also have to be worked out.

We have shown that extrusion of metals is possible with organic binders. This would be difficult since the horizontal tooling would have to accommodate the volume change and the strength during sintering may be too low as it shrinks. Therefore another extrusion method of metal powders can be used to produce minimal volume change. If a higher pressure is used, little binder and only lubricant will be need to form the green body that is at relatively high density. Direct extrusion is the typical method used and continuous pressing would be obtained with multiple sequential loading of the sample chamber. This will eliminate the pre-sinter de-binding operation and shorten the process. Also, since the green body is near net shape, little volume change will occur during sintering and the tooling/process will be simplified.

5) All aspects are very complex and interactive at each step. We have collected enough information on: (1) design of continuous microwave system for commercialization, (2) continuous extrusion process for either ceramics or metals. A continuous microwave sintering system for prototyping in Phase II leading to its commercialization has been identified. For continuous extrusion process for metals, we have identified four vendors, two in the USA and two in Europe. In the Phase II, the main R and D efforts will be focused on how to marry these two systems so that continuous fabrication and sintering of coiled tubing can be accomplished with specific properties.

**IMPROVED TUBULARS FOR BETTER ECONOMICS IN DEEP GAS WELL DRILLING USING MICROWAVE TECHNOLOGY**

## Abstract

**Technical Objectives:** The main 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, by utilizing the latest developments in the microwave materials technology. This is being accomplished by developing an efficient and economically viable continuous microwave process to sinter continuously formed/extruded steel powder for the manufacture of seamless coiled tubing and other tubular products.

The entire program has been spread over three phases with the following goals:

**Phase I:** Demonstration of the feasibility concept of continuous microwave sintering process for tubular steel products.

**Phase II:** Design, building and testing of a prototype microwave system which shall be combined with a continuous extruder for steel tubular objects.

**Phase III:** Execution of the plan for commercialization of the technology by one of the insustrial partners.

The criteria for the success of the program is based on the performance of coiled tubing made by the microwave process. It is expected that this product will have superior quality and performance to the standard product, and will be economically viable.

## Experimental

The research program has been based on the development of microwave technology to process tubular specimens of powder metals, especially steels. The existing microwave system at the Materials Research Laboratory was suitable modified to enable to process tubular specimens. The precursor powder metals were either extruded or cold isostatically pressed to form tubular specimens. The procedure for making trial samples and modifications made in the microwave systems are described below:

**i. Modification of Microwave system:** The existing vertical continuous microwave system at Materials Research Lab (MRL) has been used in the past for the processing of ceramics and WC/Co parts. To process powder metal (steel) samples the systems at MRL and Dennis Tool Company (DTC) were suitably modified by designing and installation of new insulation packages and tube assembly, etc. As the experiments were continued to be executed further modifications were required, for example the original stationary alumina tube was replaced by a larger diameter tube and the associated assembly, to facilitate the smooth movement of crucibles and long steel tubes of diameter >1.5 inch. Also some necessary modifications were made in the insulation package and microwave cavity assembly. Several brass tubes were built, joined and installed in the cavity in order to load many powder metal cylindrical samples at the same time. A photograph of this modified continuous microwave system for laboratory type experiments is shown in Figure 1.

**ii. Continuous Microwave Sintering of Trial Samples:** First, in order to develop a continuous microwave sintering of steel samples in the new modified microwave system, twelve FN208 (Fe:97.2, Ni:2.0,C:0.8) cylindrical samples (1 inch diameter and  inch height) were loaded in the microwave furnace for vertically continuous sintering. The arrangement is schematically shown in Figure 2.

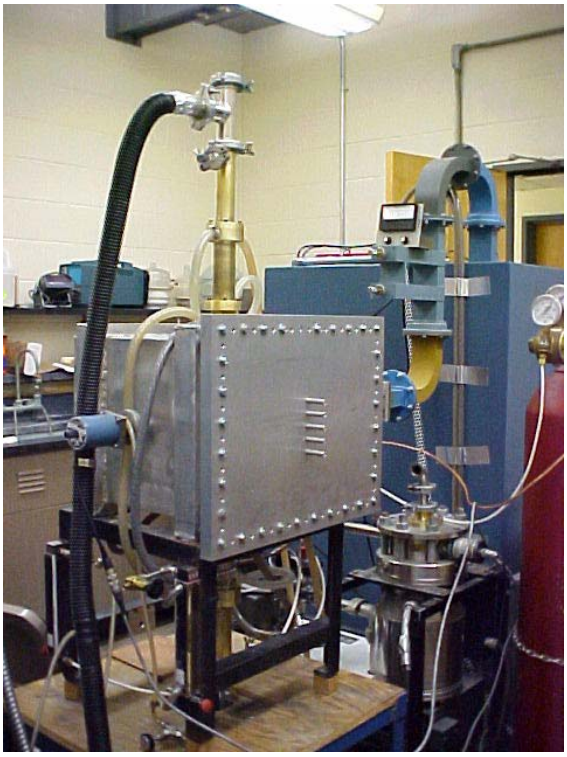
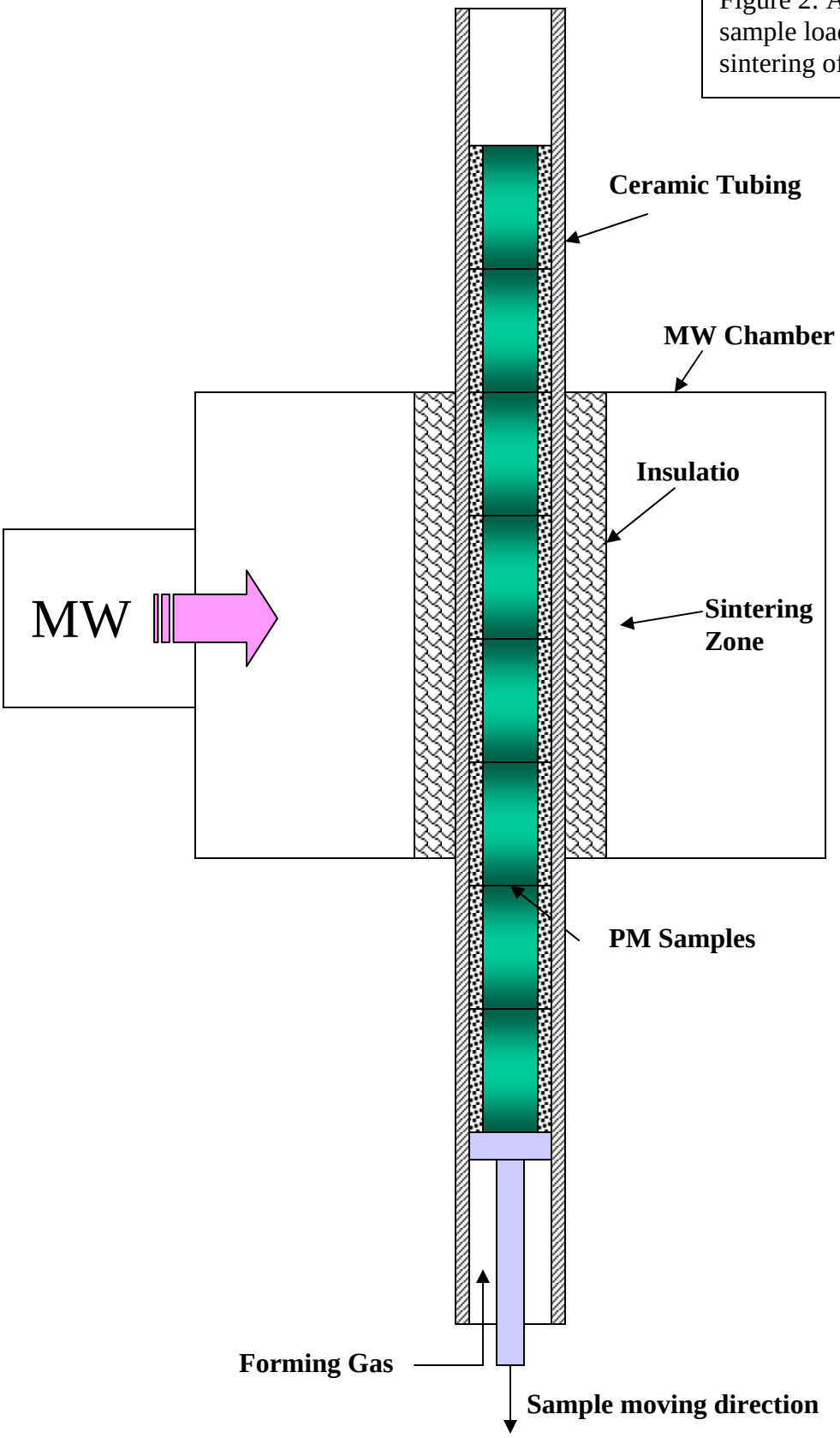


Figure 1. Continuous Microwave System modified to sinter Powder Metal Tubular Parts

The processing conditions were adjusted to obtain the optimum sintering temperature of 1250$^{o}$C for processing of FN208, at the center of the cavity. The assembly of the samples was allowed to move vertically downwards with a specific feeding rate. It was found that the softening of the bottom sample at 1250$^{o}$C and the heavy load of the top samples, caused some distortion in the shape of the sintered sample. Otherwise the samples were found to be good after sintering. So, next this experiment was repeated using alumina crucibles to separate the FN208 samples. The ceramic crucibles having much higher strength at high temperatures supported the weight of the steel samples individually. The continuous sintering of cylindrical samples was successfully accomplished with consistent and uniform sintering results. The sintering conditions used were:

Atmosphere: Forming gas (95%$N_2$+5%$H_2$), Temperature: 1250$^{o}$C, Soaking time: 5 min.
Feed rate: 0.2”/min, Total time to sinter 11 samples: 65 min.
Table 6 lists sintered density data of FN208 trial samples. It is quite clear that all samples have been sintered quite uniformly with very consistent sinter density.

Figure 2: Arrangement of sample loading for continuous sintering of cylindrical samples



**iii. Fabrication of long tubular samples by Cold Isostaical Pressing (CIP):** For fabrication of long tubular specimens both extrusion and CIP methods were adopted. CIP is a standard method for consolidation of powders by pressing uniformly in all directions. In this method uniform pressure in all sides is applied using a liquid medium and rubber bag containing the powder, providing isostatic conditions. In the extrusion method slurries of the powders are used, and subjected to push through a cavity of desired diameter providing solid/hollow tubular specimens. CIP can produce tubular samples only up to a limited length (1-2 feet). However, extrusion method can produce virtually any length of tubular objects.

While the extrusion details were being worked out, CIP was used to make 12 inch long solid and hollow tubes of nominally 1-1.5 inch diameter. Figure 3 shows CIP equipment available at MRL. It has capacity of fabricating up to 3-inch diameter and 2 foot long samples. In order to make, 1-foot long tubes the necessary molds, rubber bags and metal grid containers were acquired. CIP conditions used in this study were: 30,000psi for 5 minutes. In order to test the system, trial samples of FC208 steel powder were made. Having gained enough experience and confidence in CIP process, tubes of the chosen composition of 316 L were made. 15 test samples of about 12 inch in length and 1 inch OD using 316L steel powder mixed with 1.5 wt% binder (Acrawax) were CIP'ed. The binder burnout (removal of Acrawax) of these CIPed samples was successfully done at 450$^{o}$C in 1 hour.

**Microwave Sintering of CIPed Samples:** First for trial runs in the modified microwave system 12 inch CIP tube of FC208 as fabricated above was used for continuous sintering. It was sintered to nearly full density at 1200$^{o}$C with a feeding rate of 0.2 inch/minute. Next some process optimization was conducted on another set of tubes made by CIP. 15 CIPed specimens of 316L were used to determine the best microwave sintering conditions in flowing $H_2$, (samples were processed at

Dennis Tool Co.'s automated microwave commercial unit for cemented carbide) and sent to Quality Tubing for mechanical and corrosion resistance tests.




Figure 6: Cold Isostatic Pressing equipment used in this program to make 12 inch powder metal tubes

## Results and Discussion

### Phase I: Demonstration of the Feasibility of the Proposed Concept

***Selection, microwave sintering, testing and characterization of test samples:***

First several carbon steel compositions that are used by our industrial partner Quality Tubing (QT) for making drill tubing, were selected for microwave sintering investigation. The elemental analysis of these compositions is listed in Table 1. QT receives sheets of these steels from Mitsubishi Metal Work, Japan who makes these sheets by melting, casting and tempering by high pressure rollers into thin sheets which are cut to the required sizes for making tubular products by folding and welding method. Since these compositions were not available in powder form for Powder Metal (PM) sintering, for the current project precursor powders were made by mixing various constituting elements as listed in Table 1. Usually in PM sintering processes pre-alloyed mixes are used for consolidation. This provides high densification, uniform sintering, and better properties. Since pre-alloyed mixes of QT compositions were not available, non-alloyed pre-mixed powders prepared by Keystone Powder Metal Co. were used for initial studies.

In addition, several steel compositions were discussed with Keystone Powder Metal Company to provide green samples including QT compositions (Table 1). It was decided that in the beginning for trial runs in our existing vertical microwave system, FN208 and FC208 (Fe:97.2, Ni/Cu:2, C:0.8 wt%), standard steel compositions used for most automotive parts, should be used, until new powders were ready for the fabrication of green specimens. The purpose of these experiments was to test our existing microwave system and also to find out if any modifications were needed in the system for sintering of powder metal samples. This system was used for processing powder metal hollow and solid cylindrical samples.

Table 1: Elemental analysis of QT compositions used for coiled tubings.

| **Elements** | **QT-700** | **QT-800** | **QT-900** | **QT-1000** |
|---|---|---|---|---|
| C | 0.10/0.14 | 0.10/016 | 0.10/016 | 0.15 max |
| Mn | 0.70/0.90 | 0.70/1.00 | 0.70/1.00 | 1.65 max |
| P | 0.025 max | 0.025 max | 0.025 max | 0.025 max |
| S | 0.005 max | 0.006 max | 0.006 max | 0.005 max |
| Si | 0.30/0.50 | 0.30/0.50 | 0.30/0.50 | 0.50 max |
| Cr | 0.50/0.70 | 0.50/0.70 | 0.50/0.70 | 0.50/0.70 |
| Cu | 0.27 max | 0.27 max | 0.27 max | 0.25 max |
| Ni | 0.20 max | 0.20 max | 0.20 max | 0.20 max |
| Mo | - | 0.23 max | 0.23 max | - |
| V | - | - | - | 0.010 min |
| Nb | - | - | - | 0.005 min |
| N | - | - | - | 0.020 min |

70 green cylindrical hollow and solid samples (about 1-1.5 inch length, 1 inch dia, and 0.1 inch wall thickness) of FC208 and FN208 were fabricated by cold pressing for trial runs. Some of these samples were processed individually under various temperaturse and times conditions in flowing forming gas atmosphere (95%$N_2$+5%$H_2$). The purpose of using forming gas is to provide reducing atmosphere to remove any oxide layer on metal powders and prevent their oxidation. Forming gas is preferred because of it is inflammable. After carefully fine-tuning and optimization of the processing conditions, excellent sintered samples of both tubular and solid cylindrical shapes were obtained. Once it was established that in our existing 2.45 GHz, multimode, 6 kW microwave system standard steel powders could be sintered to high density, 50 green pressed samples (solid and hollow cylinders) of QT900 composition using pre-mixed powders were procured. This is a standard composition used by Quality Tubing for making coiled tubing by the conventional process. Table 2 gives sintering conditions and density data of some these samples. All samples were

processed in forming gas (95%$N_2$+5%$H_2$). Some of the QT900 green and sintered samples are shown in Figure 4.

Table 2: Sintering conditions and density of microwave processed QT900 tubular samples

| Sample | Sintering Conditions | Density ($g/cm^3$) | |
|---|---|---|---|
| | | Water Immersion | Geometrical |
| K950 (Cylindrical) | Green | | 6.81 |
| | 1200ºC x 10min. | 7.49 | 6.72 |
| | 1250ºC x 10min. | 7.48 | 6.71 |
| M531 (Solid Rod) | Green | | 6.75 |
| | 1250ºC x 10min. | 7.24 | 6.73 |




Fig. 4 QT900 solid and hollow cylindrical samples microwave sintered in the modified continuous microwave sintering system

The samples after microwave sintering under above conditions could not achieve full densification. Since this steel composition was not reported to have sintered via powder metal sintering method, there was no data available on its sinterability. So a systematic study to identify the optimum sintering conditions for QT900 was conducted. For this purpose small discs were

fabricated by uniaxial pressing, which were microwave sintered at different temperatures and time conditions under various sintering atmospheres. The goal of this study was to obtain nearly full density with no open or surface porosity. Table 3 summarizes the results of this study:

Table 3: Microwave Sintering conditions and density of QT900

| **Sample No.** | **Sintering Condition** | **Density ($g/cm^3$)** |
|---|---|---|
| 1 | 1150˚C/10min. $H_2$=5%, $N_2$=95% | 6.87 |
| 2 | 1200˚C/10min. $H_2$=5%, $N_2$=95% | 6.88 |
| 3 | 1220˚C/10min. $H_2$=5%, $N_2$=95% | 6.76 |
| 4 | 1225˚C/10min. $H_2$=5%, $N_2$=95% | 6.67 |
| 5 | 1270˚C/10min. $H_2$=5%, $N_2$=95% | 6.77 |
| 6 | 1350˚C/10min. $H_2$=5%, $N_2$=95% | Partially Melted |
| 7 | 1200˚C/10min. $H_2$=25%, $N_2$=75% | 6.62 |
| 8 | 1200˚C/10min. $H_2$=50%, $N_2$=50% | 6.72 |
| 9 | 1200˚C/10min. $H_2$=75%, $N_2$=25% | 6.91 |
| 10 | 1200˚C/10min. $H_2$=100% | 6.90 |
| 11 | Green | 6.75 |

These results show that atmosphere is critical for obtaining good sintering. The best results were obtained with pure or 75% $H_2$ atmosphere. Still full densification has not been achieved. The theoretical density of this steel composition is 7.5 g/cc. Few more samples were sintered at higher than 1200$^o$C in pure hydrogen, but no substantial increase in the density was achieved. Therefore, it was decided to look for other steel compositions for further studies.

Since the pre-alloyed starting powder of Quality Tubing based composition for coiled tubing was not available for sintering purposes. This composition is available only in the form of ingots or flat sheets. The powder of this composition acquired from a powder metal vendor was not pre-alloyed. The pre-alloyed powder for test samples was not possible to obtain in small quantities. And as found above the non-alloyed pre-mixed powder of QT compositions did not result in to good sinterability, a few new steel compositions which were known to exhibit high strength and high corrosion resistance characteristics, were selected for further investigation. For that reason

high chrome based compositions were selected for further studies. QT has already been investigating these types of conventionally manufactured steels (Ref. 1).

The following four compositions have been selected based on their reported properties (Ref. 2):

304L: Basic Austenitic Grade. This is the most economical of the austenitic grades and is used where material cost is a large percentage of the total manufacturing cost. It has better corrosion resistance than 303L, with good machinability. It is also non-magnetic

316L: Standard Austenitic Grade. This stainless alloy with molybdenum offers better corrosion resistance and machinability than 304L. With careful processing it can meet the corrosion resistance requirements of the more demanding applications. It also is a non-magnetic grade.

316LSC: Enhanced Corrosion Resistance version of 316L. This is a tin-modified version of 316L, having similar characteristics, but even greater corrosion resistance.

SS-100: Super Premium Austenitic Grade. This highly alloyed austenitic grade is superior to all other grades of P/M stainless steel in corrosion resistance. Its corrosion resistance is equal to that of wrought stainless 316L when formed and sintered under optimum conditions. Under less than optimum conditions, it is still superior to all other P/M stainless grades. It is also non-magnetic.

The above listed steel powders of –100mesh grade were acquired from commercial vendors. A microwave sintering study to identify the best sintering conditions was conducted. The Table 4 below lists the sintering conditions and the obtained densities of these samples. Three different starting grain sizes of the steels were prepared by sieving the as-received powders through 635, 325 and 100 mesh sieves. This was done to identify the optimum grain size which could produce best density values. The sintering temperatures used were $1325^{o}$ and $1310^{o}$C for 20 minutes. All samples were sintered in pure $H_2$ atmosphere.

Table 4. Microwave sintering conditions and density data of 4 steel compositions

| Sintering Conditions | Samples | Densities ($g/cm^3$) |
|---|---|---|
| 1325ºC/20min. (in pure $H_2$) | 316L, - 100 mesh | 7.39 |
| | 316L, - 325 mesh | 7.31 |
| | 316L, - 635 mesh | 7.43 |
| | 316LSC, - 100 mesh | 7.33 |
| | 316LSC, - 325 mesh | 7.32 |
| | 304L, - 100 mesh | 7.32 |
| | 304L, - 325 mesh | 7.23 |
| | SS-100, - 100 mesh | 7.30 |
| | SS-100. - 325 mesh | 7.45 |
| 1310ºC/20min. (in pure $H_2$) | 316L, - 100 mesh | 7.35 |
| | 316L, - 325 mesh | 7.26 |
| | 316L, - 635 mesh | 7.35 |
| | 316LSC, - 100 mesh | 7.26 |
| | 316LSC, - 325 mesh | 7.34 |
| | 304L, - 100 mesh | 7.32 |
| | 304L, - 325 mesh | 7.20 |
| | SS-100, - 100 mesh | 7.26 |
| | SS-100. - 325 mesh | 7.36 |

Based on the further characterization of the sintered samples of these four compositions, 316L was found to be the best and was selected as the final composition for scale-up experiments. Several grades of 316L powders were acquired from various sources and an investigation of sinterability vs grain size was carried out.

Metal powders of the most common metals are normally made by melting metal ore/scrap and then atomizing the melt by spraying it into a water bath or flowing gas stream. The powders are then processed and classified into the appropriate grades/size. The starting grain size is recognized to play important role in many sintering methods because the performance of the finished product is highly dependent upon the sintered density and microstructure. It is general believed that the finer microstructure provides better mechanical properties.

As for as particle size of 316L steel powder is concerned, not much difference was found in the sintered density when processed between 1300-1325$^{o}$C for 15 minutes in pure $H_2$. Several experiments were performed to sinter 316L under varying processing conditions as listed in Table 5.

Table 5: Microwave Sintering conditions and densities of the sintered samples of 316L.

| No. | Sintering Condition | Density (g/cm$^3$) |
|---|---|---|
| 1 | 1300˚C/10min. in $H_2$ | 7.03 |
| 2 | 1300˚C/20min. in $H_2$ | 7.16 |
| 3 | 1300˚C/30min. in $H_2$ | 7.41 |
| 4 | 1325˚C/10min. in $H_2$ | 7.35 |
| 5 | 1325˚C/20min. in $H_2$ | 7.06* |
| 6 | 1325˚C/30min. in $H_2$ | 7.82 |
| 7 | 1340˚C/10min. in $H_2$ | 7.83 |
| 8 | 1350˚C/10min. in $H_2$ | 7.84 |

The above results show that nearly full density was achieved at 1340$^{o}$C/10 min. The microstructural data of the above samples are quite consistent with the density results. Typical microstructures of sintered samples are shown in Figure 5. The microstructures of the sample sintered at 1340$^{o}$C/10 min show very little porosity, it is just like the microstructure observed in QT900 conventional sample received from Quality Tubing (see Figure 10) and manufactured from metal strip.

Figure 5: Microstructure of 316 L microwave sintered (a) 1300$^{o}$C, 10 min., (b) 1340$^{o}$C, 10 min.



**i. Continuous Microwave Sintering of Trial Samples:** First, in order to develop a continuous microwave sintering of steel samples in the new modified microwave system, twelve FN208 (Fe:97.2, Ni:2.0,C:0.8) cylindrical samples (1 inch diameter and  inch height) were loaded in the microwave furnace for vertically continuous sintering as described in Experimental section. Table 6 lists sintered density data of FN208 trial samples. It is quite clear that all samples have been sintered quite uniformly with very consistent sinter density.

Table 6**:** Sintered density of FN208 samples continuously sintered in microwave

| Sample No. | Density ($g/cm^3$) |
|---|---|
| 1 | 7.36 |
| 2 | 7.36 |
| 3 | 7.34 |
| 4 | 7.32 |
| 5 | 7.32 |
| 6 | 7.32 |
| 7 | 7.32 |
| 8 | 7.34 |
| 9 | 7.34 |
| 10 | 7.32 |
| 11 | 7.34 |

Figures 6 shows the continuously sintered FN208 samples.




Figure 6. Eleven FN208 samples microwave sintered continuously in 60 minutes at 1200C (a) hollow cylinders (b) solid cylinders

**ii. Cold Isostatically Pressed samples sintered in microwave:** Figure 7 shows 12 inch CIPed tubes of FC208. After having gained enough experience and confidence in CIP process, tubes of the chosen composition of 316 L were made. 15 test samples of about 12 inch in length and 1 inch OD using 316L steel powder mixed with 1.5 wt% binder (Acrawax) were CIP'ed. Figure 8 exhibits 5 of such green samples.

For trial runs in the modified microwave system 12 inch CIPed tube of FC208 as fabricated above was used for continuous sintering. It was sintered to nearly full density at 1200$^{o}$C with a feeding rate of 0.2 inch/minute. The sintered samples were cross-sectioned and examined under SEM. Some surface porosity was observed. Surface porosity is defined as the pores developed on the surface of a sintered product, and they are considered undesirable due to the increase in the apparent surface area and thereby making the surface more susceptible to corrosion attacks. Next some process optimization was conducted on another set of tubes made by CIP. After conducting many experiments, temperature of 1250$^{o}$C and 0.2 inch/min. feeding rate were found to be optimum for FC208. The long sintering times at temperature are critical for solid state sintering of metals. The movement rates of up to 10-minutes/inch length and temperature of up to 1305$^{o}$C were tested. At these high temperatures, melting is a risk since the liquidus temperature is close. 15 CIP specimens of 316L were used to determine the best microwave sintering conditions in flowing $H_2$, (samples were processed at DTC's automated microwave commercial unit for cemented carbide) and sent to Quality Tubing for mechanical and corrosion resistance tests. Figure 9 shows one microwave sintered 12 inch tube of 316L.

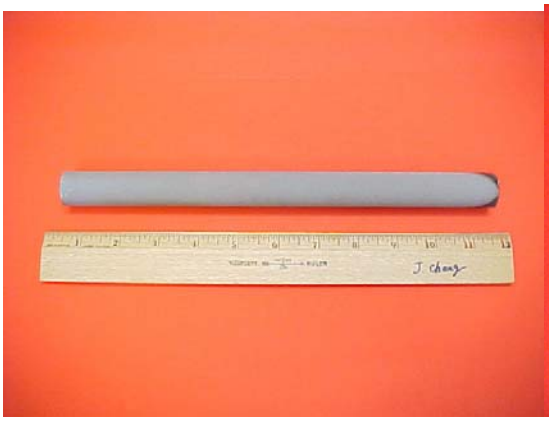


Figure 7. 12 inch long and 1 inch diameter FN208 tube continuously microwave sintered at 1200C in 55 minutes in the modified multi-mode microwave System



Figure 8: 12 inch tubular green samples 316L steel fabricated by CIP at 30,000 psi.



Figure 9: 12 inch tubular microwave sintered sample of 316L steel

Quality Tubing is conducting long range corrosion tests on the 12 inch microwave sintered tubes. They have completed preliminary examination of the microwave sintered tubes and summarized their findings in the attached letter (Attachment A). They are very enthusiastic about the progress made so far and the results obtained on the various aspects of the project.

**iii. Characterization and Testing of samples:** Several conventionally processed samples were received from QT for characterization and comparison with the microwave sintered samples. They were characterized for their density, hardness and microstructure. Table 7 lists some of the density and hardness data of these samples. The hardness values in Rockwell C were determined using a hardness tester. It should be noted that due to the mounting of the samples into the plastic the true hardness values could not be determined as plastic absorbs some of the stress from the indenter:

Table 7**:** Density and hardness values of Quality Tubing Samples

| **Sample** | **Density ($g/cm^3$)** | **Rockwell C** |
|---|---|---|
| QT 1000 | 7.83 | 22.4+0.1 |
| QT 900 | 7.84 | 15.3+0.8 |
| QT 800 | 7.84 | 16.0+0.8 |
| QT 700 | 7.82 | --- |
| HO 70 | 7.86 | --- |

*Phase Composition:* For the determination of the phase composition by XRD, five rings were cut at a place away from the seal. The cutting plane was ground, but not polished, because the polishing of steel composition results in to preferred orientation of the upper few micron layers. Since iron has an absorption edge near the Cu-$K_\alpha$ line the penetration of the x-rays into the iron is very small. It is likely that the upper layer of the sample of the order of 1-2 μm may be spoiled by polishing and thereby giving a result not representative of the whole material. Grinding, however, gives a truer description of the orientation of the iron material. Two phases exist in all samples, α-Fe (cubic) and austenite (tetragonal). α-Fe is characterized by a 100% peak at 2.01Å (110) and a 20% peak at ~ 1.43Å (200). Austenite is characterized by a 100% peak at ~ 2.08Å (101), a 44% peak at ~ 2.01Å (110), and a 11% peak at 1.43 (200). In all the samples there are peaks at ~2.08Å (small), 2.01Å (large), and at 1.43Å (small) accounting for austenite and α-Fe. Table 8 lists the intensities of the X-ray peaks and the lattice parameters of austenite calculated from the d values of these peaks, and the

lattice parameters of α-Fe calculated from the 200 peak. It might be interesting to X-ray the raw material of the 5 QT compositions and compare the results with the finished product. But unfortunately the standard materials is not manufactured from the powders but ores or scrap metal and pure raw materials of these compositions are not available.

Table 8. X-ray diffraction results of Quality Tubing compositions. Intensities (%) of X-ray peaks, austenite tetragonal lattice, and α-Fe cubic parameters.

| Sample | 2.08 Å I(%) | 2.01 Å I(%) | 1.43 Å I(%) | a (aust) Å | c (aust) Å | a (α-Fe) Å |
|---|---|---|---|---|---|---|
| QT1000 | 2.30 | 100 | 6.94 | 2.832 | 3.053 | 2.861 |
| QT900 | 3.29 | 100 | 6.36 | 2.847 | 3.050 | 2.866 |
| QT800 | 2.68 | 100 | 9.61 | 2.843 | 3.047 | 2.871 |
| QT700 | 1.60 | 100 | 5.01 | 2.850 | 3.053 | 2.870 |
| HO70 | 1.97 | 100 | 13.97 | 2.843 | 3.044 | 2.867 |
| α-Fe cubic JCPDS6-696 | | 100 | 20 | | | 2.866 |
| Aust (tetra) JCPDS44-1289 | 100 | 44 | 11 | 2.846 | 3.053 | |

*Elemental Analysis:* EDS characterization was performed on the QT900 powder prepared for microwave sintering and compared with the EDS spectra of conventional sample of the same

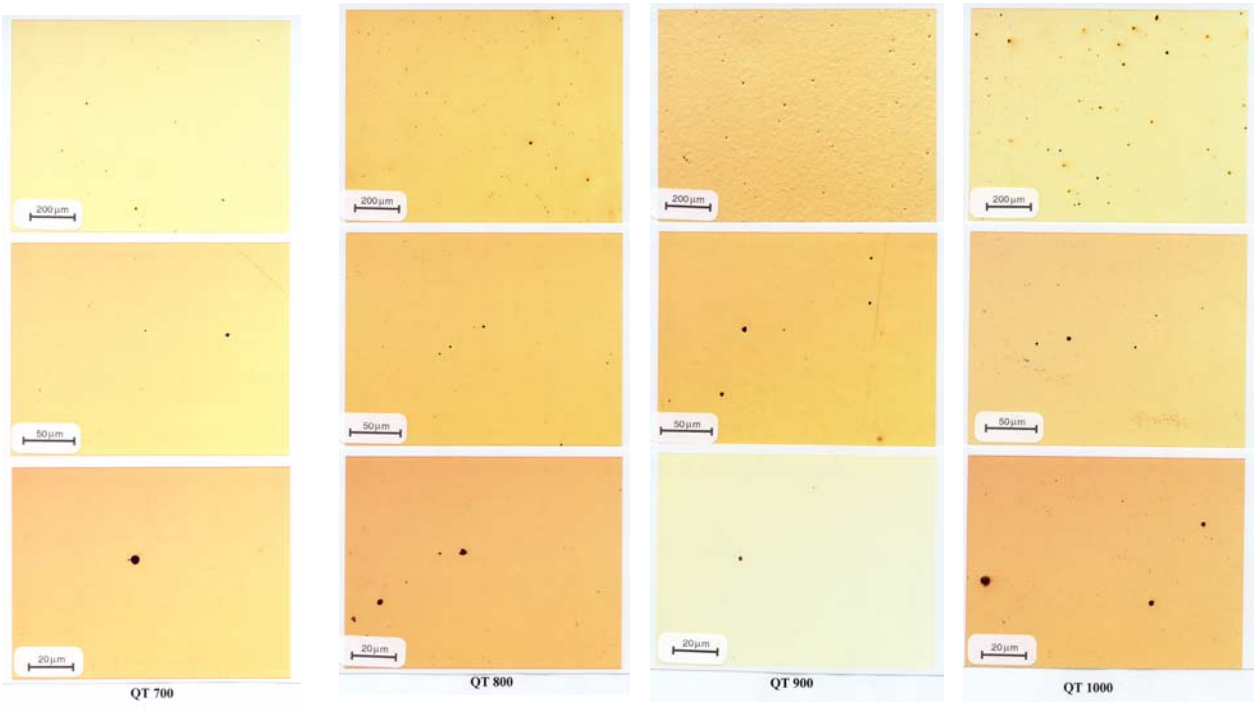


Figure 10. Microstructure of Quality Tubing conventionally pressed coiled tubing samples (QT 700, QT 800, QT 900 and QT 1000)

composition received from Quality Tubing. Other QT samples were also characterized by EDS for their elemental analysis. The EDS analysis shows that all samples have almost identical EDS spectra, and contain Fe as a major phase and Si and Cr as the minor phases. Other minor phases are below the detection limit. The results are quite consistent with the data provided by Quality Tubing (Table 1) and indicate that powders were the same as was provided by Quality Tubing for their standard steel for coiled tubing.

*Microstructure:* All five samples were re-polished with 1µm diamond paste followed by 50nm colloidal silica. This was to remove all scratches. The microstructures of the polished surfaces were examined by an optical microscope using standard metallographic techniques. Figure 10 shows some representative micrographs of these compositions. A small degree of porosity was evident in all QT samples. Even though these samples were rolled from large ingots, they still were apparently not at fully density.

**iv. Modification of 316L by adding dopants*:*** In order to continue to improve the mechanical properties and performance of the chosen 316L steel, a side-study (*at Indian Institute of Technology, Kanpur, India at no cost to the project*) was conducted to modify the composition by adding some oxide dopants to improve the strength and corrosion resistance. For this purpose few % of YAG (Yttrium aluminum garnet, $Y_3Al_5O_{12}$) or $Y_2O_3$ were added to 316L. These dopants are well known for improving the sinterability and mechanical properties of the steel compositions. The purpose of these two dopants is to eliminate the inter-porosity in 316 L and thereby enhancing the mechanical properties. So far these samples have been sintered only by conventional heating method. The microwave sintering experiments are in progress. Some preliminary data is reported hereunder indicating that there is a substantial increase in the mechanical properties of the doped 316L. Table 9 list the strength data. What we have found that there is an optimum value of 5% of YAG that enhances the strength by over 50 % over the pure 316L. Figures 11 and 12 show strength (YS- Yield Strength, UTS- Ultimate Tensile Strength) and hardness data respectively indicating

substantial improvement in the values by adding 5% YAG. Such substantial improvement in the properties was achieved just by conventional sintering method and it is believed that these values can further be improved by microwave sintering due to its inherent advantages. These values are very compatible with the strength values normally required for coiled tubing applications. Similarly, the hardness values have also substantially increased as shown in Figure 12 at 1400$^{o}$C sintering in a conventional method. This is expected to increase further by sintering in microwave field. Figure 13 displays stress-strain curve of 316L as a function of dopant addition. And here also small amount of YAG and borides improves the strength substantially. This work is being proposed to be continued through Phase II to obtain optimum composition of the modified 316L.

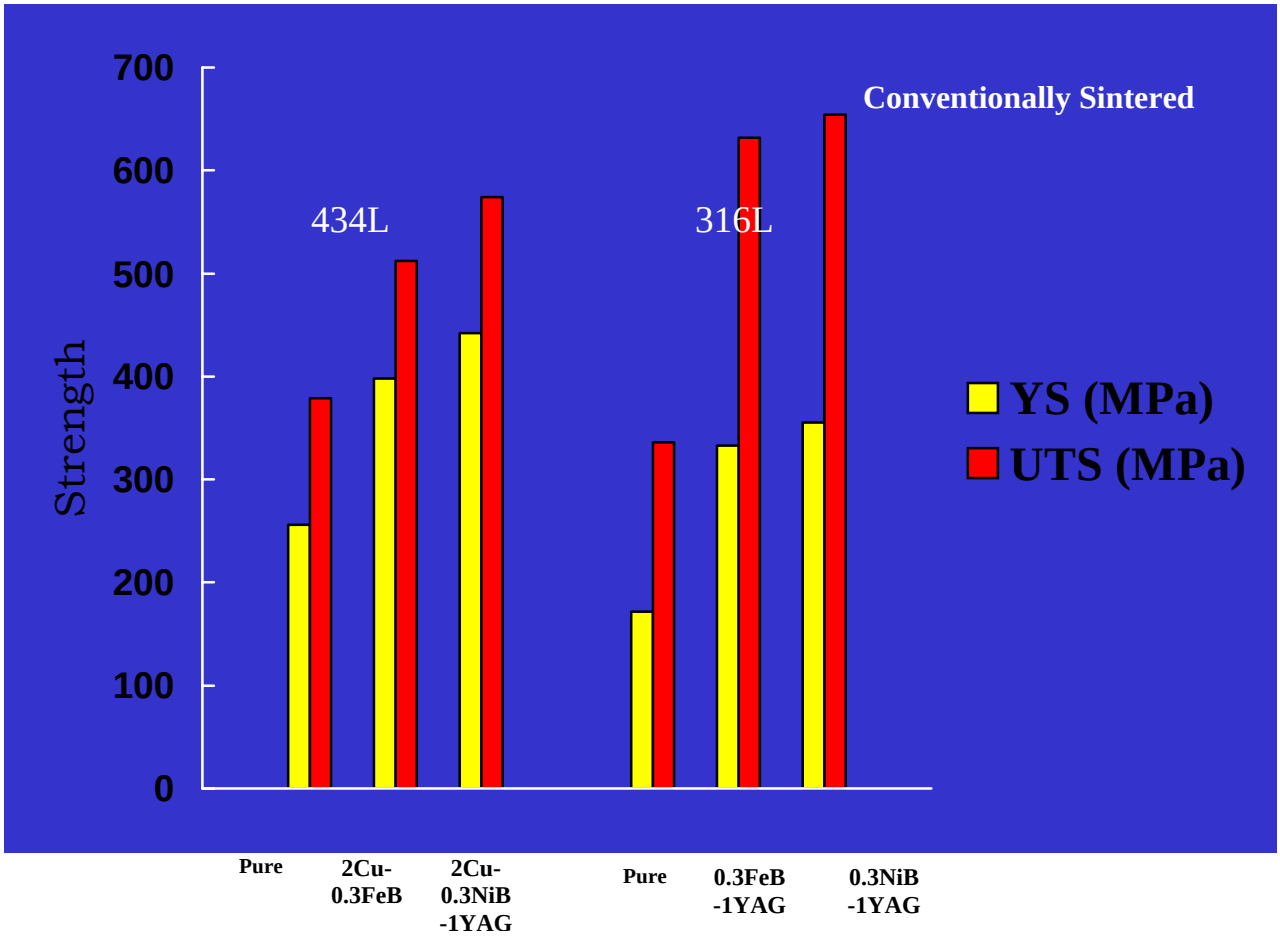



Figure 11. Variation of Tensile Properties with Composition and dopants in 316L and 434L steel conventionally processed

**Legends**:
Pure:316L,
2Cu-0.3FeB-1YAG: 2wt% Cu + 0.3wt% FeB + 1wt% YAG + 96.7wt% 316L
2Cu-0.3NiB-1YAG: 2wt% Cu + 0.3wt% NiB + 1wt% YAG + 96.7wt% 316L

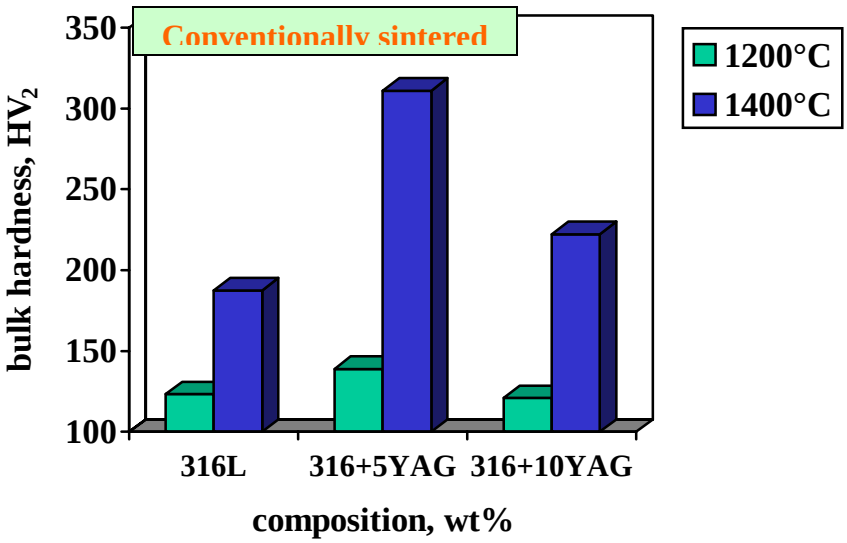



Figure 12. Comparison of hardness values of 316L and modified 316L with YAG showing 5 wt% as optimum amount

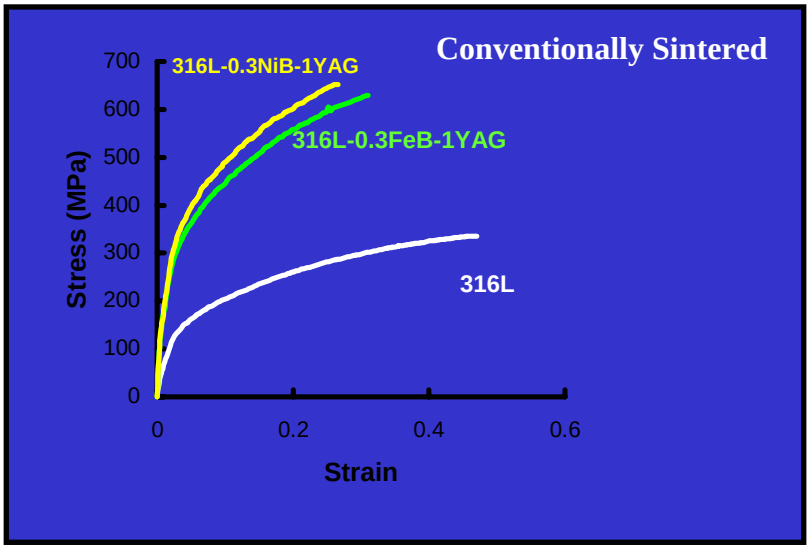



Figure 13. Stress-Strain Curve of 316L with additives

Table 9: Yield Strength (YS) and Ultimate Tensile Strength (UTS) of 316L doped with YAG as sintered by conventional heating.

| Composition wt% | Yield Strength (Mpa) | Ultimate Tensile Strength | %Elongation |
|---|---|---|---|
| 316L | 232 | 398 | 63 |
| **316+5YAG** | **265** | **420** | 27 |
| 316L+10YAG | 222 | 252 | 9 |

***Design concept of Continuous Forming/Extrusion Process for Carbon Steel Powders***

Materials Research Laboratory has a Braebender extruder, however, the equipment was not in use for over a year and hence needed some repairs and cleaning up. Non-wax binder powder was acquired since the normal wax binders are very difficult to remove in a short period of time when used in the quantities needed for extrusion. Consequently, water based binder (methylcellulose) was tested. The binder powders were mixed with water and powder metals to determine the proper concentration parameters. The goal was to determine the minimum binder plus water to obtain the proper powder rheology for extrusion into tube shapes. This includes the handling and de-binding characteristics of the green bodies. The de-binding procedure is critical. The intention was that the extruded tube should be robust enough to be introduced into the microwave applicator directly from the extruder. The sintering of pressed pellets to determine shrinkage parameters as the materials are sintered to full density was also investigated. The steel powders have been sintered to nearly full density and processing parameters such as reducing gas flow were worked out. The sintering experiments have shown that iron materials are difficult to sinter to full density. The parts tend to have more porosity at the surface as compared to the center. This appears to be due to the poor coupling of the microwave energy to the iron powders as demonstrated by the following experiments. Tests for bonding two discs of iron powder (bonding of lengths of pipe together) were not successful until a cobalt cemented carbide mix was tested. The

carbide coupled well and heated the iron discs to melting. When nickel and cobalt powders were tested in contact with the iron powder, they sintered well but the iron powders did not, and bonding was minimal. Also, 5% mixtures of the various powders in iron sintered well. A method of coating/spraying the parts with microwave susceptors in order to increase their heating at lower temperatures improved the sintering as well. A susceptor is a material which absorbs microwave energy at room temperature very efficiently, and is normally used with a workpiece which is not good microwave absorber at room temperature. Therefore, it is a general practice in microwave process to heat low absorbing materials effectively by using some susceptor such as SiC, $MoSi_2$, graphite etc.

A full range of viscosity mixes of the metal powder/binders for the extrusion of the metal powders has been tried without the formation of good parts for sintering. The Braebender extruder could not move the material through the screw and barrel without binding when very dry or with acceptable shape when moist. The methylcellulose-water binder was put on hold. In order to demonstrate the sinter-ability and extrude-ability of steel in a timely fashion, a program with the CISP (Center of Innovative Sintered Products) group at PSU was setup to produce extrudate with 316L stainless powder. The first step of this program was to adopt an organic base binder material based on “in house” technology (Ref. 3). The 316L stainless steel used for the extrusion experiments is pre-alloyed and fine (< 20 μm). The powder metal of 316L was extruded into quarter inch diameter rods, binder removed, and parts were pre-sintered. The pre-sinter step is done to add green-body strength without a volume change in order to transfer the parts to the MW system. Binder concentration was 35 wt%. The green bodies were sintered in forming gas at 1320–1360$^{o}$C with measured densities up to 7.84 g/cc. This stainless steel has a theoretical density of 7.9 g/cc. The shrinkage was about 30% volume change and the shape was maintained. There was some

variability with the density for similar processing conditions (6.87-7.84 g/cc), which is probably due to the nitrogen in the forming gas. Pure hydrogen is recommended for these compositions since chromium forms nitrides, which does not allow the metal to shrink to full density once formed. The hydrogen not only reduces any oxides on the powders but also its molecules being small can diffuse out of pores readily. The in-house capability is limited to solid rod sizes at about 1 inch diameter with the existing tooling. This would be a good size for a continuous reactor.

The existing microwave reactor was modified to accommodate the more flammable gas mixture such as $H_2$. Another gas handling line was installed for this purpose. The extruded rod was sintered with up to 100% hydrogen. The sintered parts had a more uniform porosity and density of 7.45 g/cc was obtained. The sintering time was extended to 10 minutes / 2 inch step from 7 minutes / 2 inch step that helped improve the uniformity and reduce porosity. In order to compare these results with the conventionally sintered samples, some rod samples were sintered using the normal sintering process. And the porosity results were found to be similar. Since it did not sinter to theoretical density, the powder is believed to be suspect, and is being checked with respect to its proper specifications.

**Development of design of continuous microwave sintering and extrusion/fabrication of Steel Tubing:**

The design of a process for continuously sintering the tubular materials continues to be worked out vigorously with our industrial partners and our contacts in the microwave and P/M community. The extrusion of steel into tubes will require development of unique techniques for extruding tubes that are transferred to the microwave applicator in a manner consistent with the sintering times that will be needed. The development of a de-binding method that can be matched with the extrusion times and sintering times is a challenge. A long de-binding unit and a long microwave unit will be needed to accommodate the relatively quick extrusion process and to insure a useful production rate of tubing. The maintaining of the proper de-binding/sintering atmosphere both inside and outside of the green and sintered tube will also have to be worked out.

We have shown that extrusion of metals is possible with organic binders with concentrations of 35% by weight. This would be difficult since the horizontal tooling would have to accommodate the volume change and the strength during sintering may be too low as it shrinks. Therefore another extrusion method of metal powders can be used to produce minimal volume change. At present four sources of extrusion methods are being pursued: 1. Extrusion Research and Development Center of TU, Berlin, Germany, 2. Keratech Ceramic Rollers, Romans Italy, 3. Loomi's Product Company, Pennsylvania, and 4. S.C. Starck, USA. Before deciding which of the four techniques is the best suitable for our purpose, some research and evaluation is needed. Our purpose is first to consider and fully explore the domestic sources and if found inadequate, then go for the foreign sources.

As an example: if a higher pressure is used, little binder and only lubricant will be need to form the green body that is at relatively high density. Direct extrusion is the typical method used (Figure 14) and continuous pressing would be obtained with multiple sequential loading of the sample chamber (Figure 15). The direct extrusion may require a force or temperature that is too high since wall friction in the pressing chamber may occur and indirect pressing may be required (Figure 14). The powders do not move against the chamber walls but extrude directly through the orifice as the punch with the orifice is pushed into the chamber and against the powder billet.

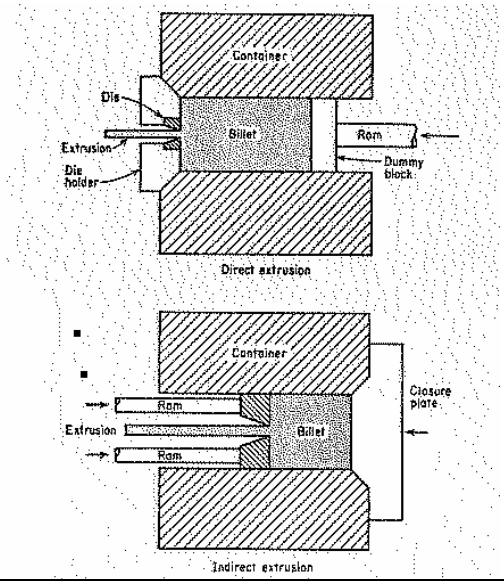



**Figure 14. Direct Extrusion and Indirect Extrusion Methods**

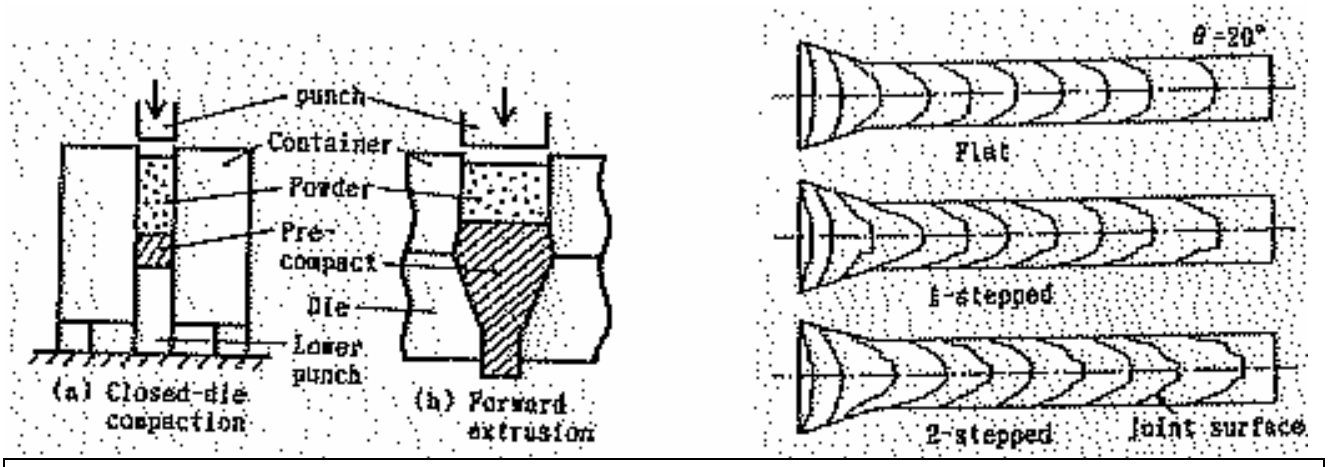



**Figure 15. Demonstration of multiple sequential loading of the sample chamber: (a) compaction method (b) joint surfaces of rods extruded with flat and stepped head punches**

These types of extrusion will be tested and the integrity of the green body will be maintained since no binder has to be removed before sintering. This will eliminate the pre-sinter de-binding operation and shorten the process. Also, since the green body is near net shape, little volume change will occur during sintering and the tooling/process will be simplified.

The current sintering operation requires feed rates of about 0.2 inches per minute in a two-foot long applicator. This is only 24 feet per day. To obtain a useful production rate the length of the MW applicator needs to be increased to about 105 feet in order to obtain about 2500 feet per day. However, with the new steel compositions, liquid phase sintering should increase the sintering rate, speed up the production rate, and reduce the applicator length.

All aspects are very complex and interactive at each step. Enough information has been collected on: (1) design of continuous microwave system for commercialization, (2) continuous extrusion process for either ceramics or metals. With our active collaboration with Japanese researchers at National Institute for Fusion Science (NIFS), a continuous microwave sintering system has been identified for prototyping in Phase II leading to its commercialization. This unit is

a 25-ft long continuous microwave-sintering kiln (Fig. 16) and is the key for the success of this project. They have also successfully made commercial continuous kilns up to 60 ft long. The details to build a similar system here or at DTC facility in the Phase II are being worked out. The collaboration between the Japanese researchers and Penn State has been approved by the Office of Fusion Energy, DOE, Gaithersberg headed by Dr. G.V. George. Under the program, visits of scientists of two institutes will be exchanged for conducting experiments in microwave processing of metals and ceramics, and also the exchange of technical information and use respective facility. Has also been allowed. So we do not see any intellectual property hurdle in the technology transfer.



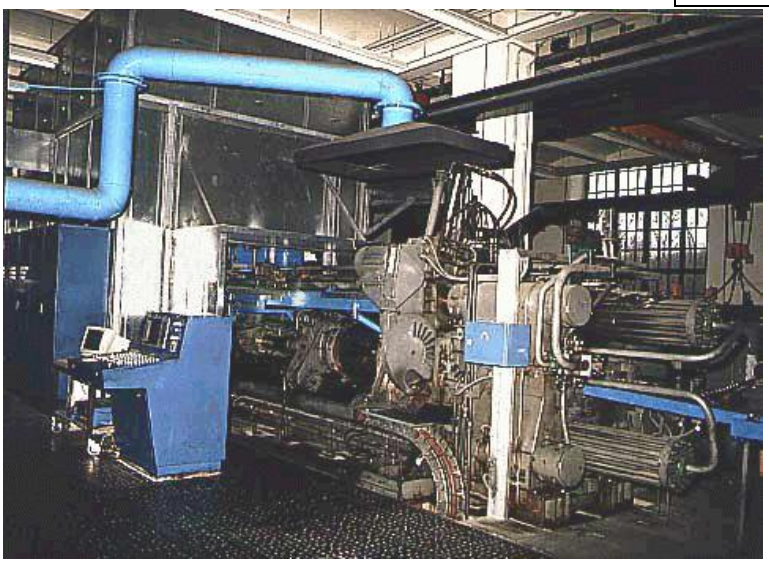

Figure 16: Continuous microwave system built by Mino Ceramic Co. for ceramics and metal products (*Courtesy: Prof. M. Sato, National Institute for Fusion Science, Japan*)

For continuous extrusion process for metals, number of vendors have been identified: two in USA, one in Germany and one in Italy. Figure 17 and 18 provide some of the details of these systems. Now in the Phase II, the main R and D efforts will be focused on how to marry these two systems so that continuous fabrication and sintering of coiled tubing can be accomplished with specific properties.

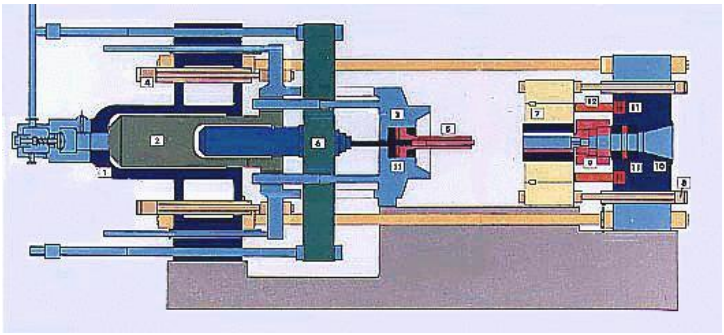
**Fig. 17: Technical data of the extruding system**

**Design:** horizontal, with piercing appliance on the inside
**Extrusion method:** directly, indirectly
**Operating pressure:** 315/250 bar
**Pressing capacity:** 8.3 MN
**Main-ram crosshead feed:** 1.11 MN
**Ram speed:** 0 to 80 mm/s, regulated
**Ram stroke:** 1180 mm
**Piercing capacity:** 1.31 MN
**Mandrel stroke with relative stroke:** 530 mm
**Container - contact pressure:** 0.79 MN
**Diameter of the container:** 85, 95, 110, 125, 140 mm (heatable)
**Billet length, max:** 500 mm
**Drive:** direct oil hydraulic
**Control:** PLC
**Billet heating:** Inductive heating
**Products:** Bars, profiles; tubes

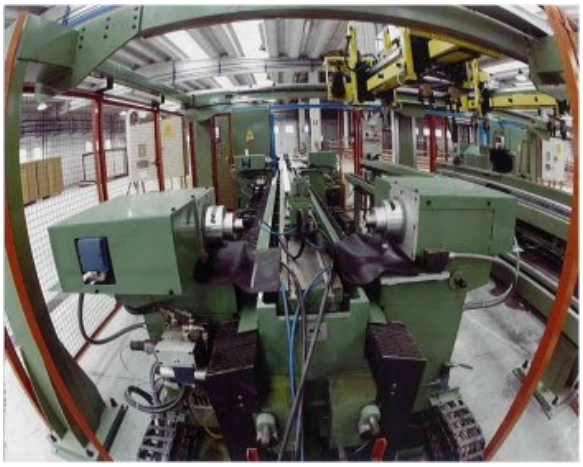
*Coutesy:* Forschungszentrum Strangpressen FZS
**Extrusion Research and Development Center of TU Berlin**



**Legend to the picture**



- Main cylinder
- Main plunger piston
- Main ram crosshead
- Main ram crosshead- feed cylinder
- Ram with mandrel
- Piercing mandrel with stroke limiter, Mandrel turning fixture and mandrel interior cooling
- Container
- Container - shifting cylinders
- Tool turning heading
- End housing
- Dynamometers
- Stilts





**Courtesy: Keratech Ceramic Rollers**
***Via 2 Giugno 7 34076 Romans Italy***

Figure 18. Extrusion equipment for Ceramic Rollers which can be modified and adopted for powder metal steel powder coiled tubing

***Preliminary Project Financing Plan***

The process consists of 1) powder manufacture, 2) powder preparation for extrusion, 3) extrusion of the powders into the green tubular bodies, 4) continuous de-binding of the green bodies, 5) microwave sintering of the tubulars, and 6) finishing/coiling of the tubulars. Each step is a major project in itself and originally all steps except 5), the microwave sintering, was thought to be current proven industrial technology.

However, only steps 1) powder manufacture, 2) powder preparation for extrusion, and 6) finishing/coiling of the tubulars are currently proven industrial technology. These can be scaled to the process without major alterations and will not be demonstrated next year. Powders can be purchased in tonnage quantities and it is feasible to setup a plant at the coil tubing manufacturing facility if powder volume justifies it. Powder preparation can also be done by the powder manufacturing facility. Finishing and coiling the tubulars are currently being done in the manufacturing of the current welded tubulars and can be adapted to the manufacture of microwave

sintered coil tubing since the tubulars will be hot as they exit the microwave applicator and this energy will be saved by processing them continuously.

Steps 3, 4, and 5 (extrusion of the powders into the green tubular bodies, continuous de-binding of the green bodies, microwave sintering of the tubulars, respectively) need to be finalized for the successful manufacture of the microwave sintered coil tubing. The continuous sintering of metal tubes has been demonstrated this year. The basic process parameters have been defined and the longer applicator is needed for a manufacturing process.

The extrusion of powder metal/binder thin rods has also been demonstrated. The de-binding has only been done in batch tests but with the binder approach it would require a long de-binding unit to insure total removal of the binder since it is a slow process. The complication is that the green tubular has to be continuously supported for this to occur. Consequently, the binder-less approach would be tested first which requires sequential ram extrusion for the continuous extrusion.

In this case, the tubulars would be extruded with only a lubricant that will be eliminated during microwave sintering without any extra handling. The volume change would also be much less in this case since there is no binder volume that has to collapse to full density. This would also eliminate a long de-binding unit that would have to be placed between the extruder and MW applicator.

The cost of the demonstration of the two relevant steps (3 and 5) would be the cost to either renting or buying/building the units. There are research and development groups already mentioned in task 4 that have been contacted to do these steps separately. However, if they are unable to do them in a timely fashion, the buying/building of each unit would be on the order of $200,000 each that would have to be taken care of as a supplemental budget. For demonstrating the

process renting the technology would be the best method before a major capital investment would be made at an industrial site. We will opt for this approach since in this there is no additional cost involvement.

The major industrial benefit of the final microwave sintering of coil tubing would be the direct manufacture of the seamless tubulars at the industrial site, the elimination of the current welding/annealing steps, reduction in the energy costs to microwave sinter as compared to ingot and rolling methods. Of course, the possibility of steel composition alterations for improved metal properties is much more feasible with powder sintering since huge melts are not needed.

As a preliminary estimate of manufacturing cost scenarios of microwave energy, the following analysis was prepared. This is based on the current cost of stainless steel powder metals, the typical sales price of the welded stainless steel coil tubing, and a sales price that is a 50 % mark up of manufacturing costs. Table 10 summarizes the results. As expected, the typical cost of raw materials (powder metals or metal strip) is about 90 % of manufacturing costs. The current fabrication methods require metal forming, welding, splicing, annealing, weld cleanup and inspection processes that are eliminated by the seamless tubing processing. These are estimated to be comparable or little higher than the extrusion and microwave sintering by the proposed process. Consequently, it is estimated that microwave processing costs would be in line or less *(taking in consideration of the price of 316L as $2.25/lb which is the highest in the last few years due to the increase in the price of Ni. If Ni price goes down the raw material cost will also go down in the same proportion*) than the current costs without including the energy savings gained by elimination of the strip processing from casting and rolling operations of the strip manufacturing operations that are included in the strip costs. At this point it is very difficult to predict an accurate cost of the microwave processed coiled tubing. Here only some rough estimates are given, however, the main

advantage using microwave is the development of seamless tubing which would provide longer life and better performance.

Table 10: An estimated cost analysis of the seamless coiled tubing using microwave technology

| Size OD | Raw Mat'ls Cost/lb | Wall Thickness | Weights per Foot | Raw PM Cost per Foot | Allowable Mfg Cost per Foot | Allowable Fab Cost | % Cost of Raw PM |
|---|---|---|---|---|---|---|---|
| (inch) | ($) | (inch) | (lb/ft) | ($/ft) | (50% Sale Price) | ($/ft) | (%) |
| 1 | 2.25 | 0.109 | 1.038 | 2.34 | 2.55 | 0.21 | 92 |
| 2 | 2.25 | 0.175 | 3.414 | 7.68 | 8.60 | 0.91 | 89 |
| 3.5 | 2.25 | 0.203 | 7.155 | 16.10 | 17.48 | 1.38 | 92 |

**Recommendations:** It is recommended that before starting the Phase II for Prototype Building of the system, a transition task as described below should be undertaken:

It is recommended that a transition step of “proof of concept” before executing the design and building of a prototype system, should be undertaken. In the transition period (about 6 months) continuous extrusion process using commercial extruders, and horizontal continuous microwave system (available with Japanese collaborator) should be used to demonstrate continuous fabrication (green tubular specimens) and continuous sintering of specimens in the size of at least 2.5 inch OD and 10 feet long.

**Conclusions:** Following accomplishments were made in the Phase I of the program:

1. Existing multimode, 2.45 GHz microwave systems were suitably modified to be able to process powder metals.
2. System was further modified to process small tubular specimens continuously to obtain uniform sintering characteristics.
3. Several steel compositions including Quality Tubing provided compositions were acquired and microwave sintered.
4. 316L was found to be the most suitable for further studies.
5. Extrusion experiments using 316L powders were carried and binder formulations were identified to obtain good sinterability.
6. Cold Isostaic Pressing method was adopted and optimum conditions were identified to fabricate green samples of 1inch ID and 12 inch long tubular specimens.

7. Continuous microwave sintering of 1 inch diameter and about 12 inch long 316L steel tubular specimens was accomplished with uniform microstructure and nearly full density.
8. 316L stainless steel powders were modified by adding dopants to improve the hardness, strength and corrosion resistance properties. This was achieved using conventional sintering process.
9. Several industrial vendors for extruding steel tubes were identified and preliminary discussions for scale-up operation or building a prototype were conducted.

**References:**


1. J.R. Martin and W.D. Arnam, "The Development of a Corrosion Resistant Coiled Tubing Product", SPE International, SPE 81721, pg 1-7, April 8-9, 2003.
2. Stainless Steel Powders, www.omgi.com
3. D.E. Alman and J.A. Hawk, "Binder-Assisted Extrusion," ASM Handbook Vol. 7 (1998), Shaping and Consolidation Technologies, pg 375-75.

# Attachment A

**Letter of support from Quality Tubing**

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***Senior Scientist, Corporate Level III for NDE and Radiation Safety Officer***
***October 31, 2003***

Quality Tubing
A Varco Company

## Microwave Sintering of Steel Tubing

To: Dr Mahlon Dennis
Dennis Tool Company
Houston

Mahlon:

First I would like to thank you for hosting the meeting of 23rd October 2003 between Mr. David Daniel (President of Quality Tubing), Mr. John Martin (Senior Vice President of Quality Tubing), myself (The Senior Scientist at Quality Tubing), and your good self. It was very enjoyable, and we feel that a lot of good progress has been made in phase one of the production of steel tubes by the microwave sinter process. It seems that many of the initial technological obstacles have been overcome, and so we look forward to getting more samples to test in the coming months.

As you are aware, we received and tested some samples for tensile results, and are currently also having our sister company, Tuboscope, perform long-term corrosion testing to the NACE and ASTM standards that are relevant to our industry. A second batch of samples is currently being tested for tensile properties both here in our own laboratory, and also at an independent metallurgical laboratory. We eagerly await even longer samples on which we can perform fatigue tests.

We see tremendous advantages to being able to make very long lengths of both our existing products, and also the newer 316L, at a cost cheaper than we can today, and at a quality level higher than can be achieved with electric seam-welded products. Both the products we make have different uses; the coiled tubing for our workstring market, and the coiled 316L for the umbilical market, which is very large, and getting larger. Recently, for example, we have seen requests for 120 km lengths of small diameter 316L tubing that will be attached to subsea line pipe and used in deep offshore well production, and we have seen this material spun into bundles and coated with high-density rubber for certain North Sea operations. This would be an excellent market for us since the products are seamless.

We are happy with the results so far, and look forward to participating more in this project in the coming year under phase 2. I have been allotted time to participate in the project, and look forward to working with you as we attempt to look at scaling up towards long production runs.

Sincerely

Roderic K Stanley

(Dr) Roderic K. Stanley