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

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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 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 three years ago and now we are in the middle of 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). 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. This report summarizes the progress made to-date in this new approach.

The final steel composition matching with the Quality tubing’s QT-16Cr80 was short listed and used for all experiments. 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 tested. This equipment is located at the Dennis Tool facility in Houston. Microwave sintering experiments for continuous processing of the CIPed tubes are under progress in order to identify the optimum conditions. There have been some reproducibility problems and we are at present working to resolve these problems.
2. Abstract

**Technical Objectives:** 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. Other goals are that after completing the feasibility study: (i) optimum sintering conditions shall be identified, (ii) a plan for the commercialization of the technology shall be developed and submitted, and (iii) a cost analysis of the new technology and economic viability with respect to the existing technology shall be made. The intent of this project is to demonstrate that performance of the coiled tubing made by the microwave process has commercial potential and offers superior quality and performance to anything currently existing in the industry.
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.

i. Bonding of sintered rods using microwave heating: The first bonding tests were done with tubing but this configuration was found to be too difficult to work with in the beginning. Tooling and powder rings will have to be developed in order to bond these correctly and successfully. To obtain representative bonding data in order to select the strongest bond, parts in rod shapes were either microwave sintered or commercially obtained for the tensile tests. Preliminary tests of foil vs powder for bonding have indicated that the foils do not couple very well to the microwave energy, as expected. Two approaches for bonding were adopted: (i) One-step method in which green parts with bonding powders were sintered and tested to obtain long coils. The approach was not successful since the bonding and sintering temperatures are different and when processed at a single temperature, it did not produce satisfactory bonding. (ii) Two-step process in which pre-sintered samples were joined using braze powder. For this a fixture was developed to assure alignment and apply force on the rods/bond during sintering. Four powder metals (50:50 Fe:Ni, 58:42 Fe:Ni, Co, Nitronic 30) were tested for identifying the right braze powder to produce highest bonding strength. After the initial manual lab strength tests by the operator, the mechanical testing was done on representative bonds using Instron machine and standard procedure. 50:50 Fe:Ni powder exhibited highest tensile strength of 54,313 psi which is quite a moderate value. This was obtained without any optimization of the processing conditions. The graphs of the stress data are shown in Figures 1-3. Further experiments were made 50:50 Fe:Ni powder to optimize the brazing conditions. After the surfaces were machined to a uniform diameter, the bonded rods with the 50:50 Fe:Ni powder had the highest tensile strength of 72,821 psi. This value is comparable with the conventional strength.
We also prepared more samples using other brazes and commercial Nitronic 50 stainless steel. The tensile tests were made with the Nitronic 50 stainless steel rod but with typical metal/carbide high temperature/strength brazes. Using Dennis Tool Company braze technology, the rods were bonded with cobalt copper manganese and nickel copper manganese alloys. The results are shown in the table below:

**Table 1:** Tensile test results of the Brazed samples using various commercial powders

<table>
<thead>
<tr>
<th>Sample</th>
<th>P&lt;sub&gt;max&lt;/sub&gt; kN</th>
<th>OD (mm)</th>
<th>area (mm&lt;sup&gt;2&lt;/sup&gt;)</th>
<th>Stress (max)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CoCuMn#1</td>
<td>31.07</td>
<td>12.12</td>
<td>115.37</td>
<td>269</td>
</tr>
<tr>
<td>CoCuMn#2</td>
<td>27.00</td>
<td>12.11</td>
<td>115.18</td>
<td>234</td>
</tr>
<tr>
<td>NiCuMn#1</td>
<td>72.96</td>
<td>12.11</td>
<td>115.18</td>
<td>633</td>
</tr>
<tr>
<td>NiCuMn#2</td>
<td>42.56</td>
<td>12.12</td>
<td>115.37</td>
<td>369</td>
</tr>
<tr>
<td>Standard</td>
<td>95.88</td>
<td>12.10</td>
<td>114.99</td>
<td>834</td>
</tr>
</tbody>
</table>

The standard is the tensile test of Nitronic 50 rod without any bond, which compares favorably with the literature result of 124 ksi for annealed Nitronic 50 steel. This confirms that the testing procedure used here is accurate. The result of NiCuMn#1 showed a tensile stress of about 92 ksi which is excellent when compared with the standard value of the Quality Tubing’s QT-16Cr80 (Coil-13941) composition.
Figure 1. Tensile Stress data (stress and extension) of bonded sample using 50:50 Fe:Ni.

Figure 2. Tensile Stress data (stress and extension) of bonded sample using Co.
Figure 3. Tensile Stress data (stress and extension) of bonded sample using 58:42 Fe:Ni

ii. Cold Isostatic Press (CIP) Equipment: Since CIP processing has been defined as the method for manufacturing the green samples. Two types of CIP equipment are available. Wet bag and dry bag units could be applied to this project. Dry bag is usually automated and done when large numbers of parts are needed. This would be considered for the final production equipment but would be very costly and time consuming for this stage of the development. Consequently, wet bag was considered for our work for now. New machines were two to three times as expensive as the used ones, so the latter is being looked at first. We tested a small CIP machine (4” ID, 18” long, 60,000 psi) at Flow Autoclave (Columbus, Ohio). The results were highly satisfactory with CIPed samples obtained and microwave sintered. Figure 4 shows a CIPed tube at 60,000 psi and then sintered at 1250°C for 15 minutes in microwave.

After visiting two CIP manufacturers, Flow Autoclave and American Isostatic Press (AIP), both located in Columbus, Ohio and testing their equipment and considering the price etc. finally we purchased a refurbished large CIP system from AIP (Figure 5). This unit is now installed at Dennis Tool in Houston.
Figure 4. Green CIPed samples (left) and microwave sintered at 1250°C/15 min (right)
Figure 5: Cold Isostatic Press from American Isostaic Press to be used for making green tubes.
4. Results and Discussion

Microwave Sintering Methods and Materials Selection: Tests of different steel powders were undertaken to determine if a unique composition or method was best for microwave sintering. Steels such as 316L (normal and fine particle size), Ultra 316L (Cu and Sn additives), 17-4 PH, Mixes (316L plus Cu, Nitronic 30 PSU mix) were all tested to obtain high-density parts. We also tried to reproduce Quality Tubing’s QT-16Cr80 (Coil-13941) composition by mixing/milling various powders. The composition of the new powder was prepared by mixing 91.32% 316L stainless steel powder, 8.0 % Mn powder, 0.68% Cu powder and 1.5% Acrawax. Table 2 compares this composition with that of the Quality Tubing and it is very close.

Table 2: Comparison of the composition of QT-16Cr80 and new powder prepared.

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Si</th>
<th>Cr</th>
<th>Cu</th>
<th>Ni</th>
<th>Mo</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coil-13941</td>
<td>0.026</td>
<td>8.68</td>
<td>0.022</td>
<td>0.001</td>
<td>0.32</td>
<td>16.30</td>
<td>0.74</td>
<td>2.29</td>
<td>0.06</td>
<td></td>
</tr>
<tr>
<td>New Powder</td>
<td>0.019</td>
<td>8.09</td>
<td>0.0082</td>
<td>0.885</td>
<td>15.01</td>
<td>0.69</td>
<td>12.36</td>
<td>2.02</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

ANP (Activated Nanotech Process) powders were used to obtain full sintered density on a number of normal steel powders. Now we are working with the new steel composition (QT-13Cr80) and making test samples from this powder.

The continuous microwave sintering conditions (time and temperature) were varied from 2.5 minutes to 10 minutes per 2” crucible and 1250 to 1380°C. Faster movements of the tube appears to be the best condition since high temperatures for a shorter period of time do not distort the parts as much and sintering to full density occurs. This would also be beneficial for the overall-processing scheme since faster movement of the tubing will decrease processing costs and match the bonding/coiling process. A redesign of the continuous microwave sintering applicator was done to accommodate the larger tube size (1” vs ¾” OD) by installing larger entrance tube, gas tight attachment flange, drive mechanism, and exit chamber/door.

The continuous MW sintering conditions (time and temperature) were further investigated. The 12” tubes were pressed, stacked and sintered in separate crucibles in the
continuous applicator. The results were variable with some parts sintering to non-uniform density. The power and temperature control need to be optimized to produce uniform parts. The parts need to remain close together during sintering since the ends tend to melt as the part shrinks away from the next part, which is in a separate crucible. The first run with the parts touching as the movement through the applicator was much more uniform and is being reproduced at lower temperatures since it was sintered near the melting temperature. A new insulation material to be used for uniform temperature has been ordered and will be used to provide uniform temperature and reproducibility.
5. Conclusions

This program has a potential for the development of a novel and innovative technology for the oil and gas industries at a lower cost and better product. The microwave sintering process has been well recognized in academia and industry as faster, better and cheaper. However, until recently these benefits could not be translated into commercial products due to certain limitations of the technology, especially in a scale-up operation. But in the last several years certain developments and successes achieved at Penn State in collaboration with Dennis Tool Company in this field, have taken this technology to the market place, especially in the carbide industry for making carbide inserts and wear parts used in drilling operations for gas and oil exploration. Further, the potential of microwave sintering of metals and steels was unheard of and unthinkable until a few years ago. Now it is a reality that all metals and alloys can be sintered very effectively and efficiently in a microwave field.

Our preliminary experiments have clearly demonstrated that microwave produced steel samples have superior mechanical properties than the conventional parts. In case of the drill-pipes, the elimination of the weld seam removes the weakest point in the current system. Leakage and tube fatigue occurs at this point in welded tubing. Also, microwave processing improves the fatigue life and flexibility of the powder metal coils. Fatigue occurs when the coil is rolled and re-rolled multiple times during its use on the drill rig. Higher toughness will allow the tubing to withstand higher internal and external pressures. Finally, the microwave processing improves corrosion resistance of materials, and therefore downhole in the hostile corrosive environment microwave processed tubing would last much longer than the current tubing. If this research program is successfully completed, it will translate into substantial savings in cost and energy for the gas and oil industries.

In Phase II the microwave/induction joining of steel tubulars has been successfully achieved. It is believed that it will produce iso-compositional joints without any stresses in the main body of the tubing because the time of joining is too short and only the braze powder in microwave gets heated, therefore there is no possibility of developing undesired stresses next to the joint location.