

DOE/CE--0256-1

DE90 003277

STEEL INITIATIVE

ANNUAL PROGRESS REPORT

DEVELOPMENT OF SUPERPLASTIC STEEL PROCESSING



Office of Industrial Programs
Office of Conservation and Renewable Energy
U.S. Department of Energy
Washington, DC 20585

MASTER

September 1989

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**FIRST ANNUAL PROGRESS REPORT
ON
DEVELOPMENT OF SUPERPLASTIC STEEL PROCESSING**

**A JOINT R&D EFFORT BETWEEN INDUSTRY AND
A NATIONAL LABORATORY**

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March 15, 1988 - March 15, 1989

ABSTRACT

The influence of aluminum content and thermomechanical processing steps on developing fine microstructures in ultrahigh carbon steels (UHCS) was examined. The presence of aluminum results in ultrafine pearlitic structures without proeutectoid carbide networks, and greatly retards the eutectoid transformation kinetics. This retardation facilitates processing, which involves various combinations of DET and DETWAD steps. By applying the transformation kinetics as a guide, uniform through-thickness microstructures and superplastic properties are readily obtainable in thick sections. Superplasticity studies revealed that superplastic behavior is enhanced by aluminum additions and by increasing the amount of warm deformation during DETWAD processing. This trend is attributed to a decrease in ferrite grain size and an increase in the ferrite-grain-boundary angle, and hence, to an increase in the ease of grain-boundary sliding. Studies on the cold-rolling characteristics of a UHCS-Al alloy reveal that it can be cold-rolled to 50% in the fully pearlitic condition without annealing, and to over 60% in the fully spheroidized condition. Room-temperature tensile properties were obtained on DETWAD-processed material. Identical properties were obtained in the rolling and transverse directions, which is attributed to the ultrafine, isotropic microstructure. Two trial strand-casting runs were initiated. Modified Fe-C diagrams incorporating aluminum and/or silicon additions were constructed. Materials have been processed both at LLNL and at Ladish for evaluation of their die-filling capability. Additional UHCS alloys, which are expected to develop higher strain rates and lower flow stresses in the superplastic range than those obtained with the current UHCS-Al and UHCS-Si alloys, are being evaluated.

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GLOSSARY

DET	-	Divorced eutectoid transformation.
DETWAD	-	Divorced eutectoid transformation with associated deformation.
DTA	-	Differential thermal analysis.
A ₁	-	Temperature boundary representing the start of transformation of ferrite-plus-carbide to austenite in UHCS alloys. (Boundary between the two-phase, ferrite-plus-carbide, and the three-phase, ferrite-plus-carbide-plus-austenite, regions.)
A _{cm}	-	Temperature boundary between the austenite-plus-carbide and the austenite regions.
SRC	-	Strain-rate-change (test).
SP	-	Superplastic.
WW	-	Warm worked or warm working.
UHCS	-	Ultrahigh-carbon steel.
m	-	Strain-rate-sensitivity exponent.
HWW	-	Hot and warm working.
TTT	-	Temperature-time-transformation.
LLNL	-	Lawrence Livermore National Laboratory

EXECUTIVE SUMMARY

The influence of aluminum content and thermomechanical processing steps on developing fine microstructures in ultrahigh carbon steels (UHCS) was examined. It was shown that ultrafine pearlitic structures, containing no proeutectoid carbide networks, can be obtained by aluminum additions. Hot-working (HW) followed by slow cooling from the austenite region (above A_{cm}) will lead to a pearlitic structure free from carbide networks. Spheroidized structures, with the elimination of any pre-existing carbide networks, can be obtained either by a thermomechanical treatment or by a thermal treatment alone. These are: (1) warm working (WW), involving a divorced-eutectoid transformation with associated deformation (DETWAD), and (2) heat treating, resulting in a divorced-eutectoid transformation (DET), which requires heating to a low austenitizing temperature followed by isothermal transformation just below the A_1 temperature. The presence of aluminum greatly retards the eutectoid transformation kinetics. This retardation provides a wide window of time and temperature for processing involving various combinations of DET and DETWAD steps.

Modified processing schedules may be required for thick sections to avoid the formation of non-uniform microstructures. Such microstructures arise from incomplete spheroidization (residual pearlite) in localized regions. This results in a corresponding reduction in the superplastic (SP) properties in these regions, based on strain-rate-change (SRC) tests. By applying the predetermined transformation kinetics as a guide, uniform through-thickness microstructures and SP properties are readily obtainable in thick sections. Processed thicknesses of up to 40 mm (1.6 in.) were evaluated. Results of superplasticity studies on the UHCS-Al alloys revealed that superplastic behavior is enhanced by aluminum additions and by increasing the amount of warm deformation during DETWAD processing. This trend is attributed to a decrease in ferrite grain size and an increase in the ferrite-grain-boundary angle, and hence, to an increase in the ease of grain-boundary sliding. Further improvement can be obtained by an additional final warm-working (WW) step after either the DET or DETWAD processing.

Preliminary studies on the cold-rolling characteristics of the UHCS-Al alloys reveal that they can be cold-rolled to 50% in the fully pearlitic condition without annealing, and to over 60% in the fully spheroidized condition. Room-temperature tensile properties were obtained on DETWAD-processed material (1) after annealing at 650°C, (2) after cold rolling 50%, and (3) after austenitizing to form proeutectoid spheroidal carbides and pearlite. Identical properties were obtained in the rolling and transverse directions; this is attributed to the ultrafine isotropic microstructure. Typical yield strengths obtained for the three conditions were 860, 1,170, and 755 MPa (125, 170, and 110 ksi) with corresponding elongations of 21, 12, and 12%, respectively.

Two attempts at continuous casting proved to be unsuccessful in that premature separation of the partially solidified billet (shell) from the starter bar occurred in both attempts. The changes made prior to the second heat, however, did result in very clean steel. In both heats, the desired UHCS-Al compositions were obtained. In conjunction with these castings, some differential thermal analysis (DTA) data and an extensive amount of dilatometric data have been obtained. Analyses of these data and of the casting shell, suggest that correcting the mold taper could be a significant factor in achieving a successful casting.

Critical temperatures were obtained from dilatometric data and from hardness data of quenched samples for a large number of UHCS compositions. These data together with information gained from DTA and from cooling curves provided input for construction of modified Fe-C diagrams incorporating aluminum and/or silicon additions. Of particular interest is establishing the location of a three-phase ferrite-plus-austenite-plus-carbide region for optimizing the SP behavior in these UHCS alloys and for establishing the solidus temperature for continuous casting.

Materials have been processed both at LLNL and at Ladish for evaluation of their die-filling capability. Disks machined from these materials will be superplastically press-forged into a complex shape. Additional UHCS alloys, which are expected to develop higher SP strain rates and lower SP flow stresses than those obtained with the current UHCS-Al and UHCS-Si alloys, are being evaluated.

1. PROJECT BACKGROUND

The Lawrence Livermore National Laboratory (LLNL), North Star Steel Company, Ladish Co., Inc., and Caterpillar Inc. are jointly involved in a project to develop the technology and demonstrate the feasibility for the commercialization of ultrahigh-carbon steels (UHCS). These steels are unique in that they have superplastic (SP) properties in the warm-working (WW) range. The project is based on the guidelines given in the Department Of Energy (DOE) Steel Initiative Management Plan. The project was initiated in March 1988. The current effort at LLNL is funded by DOE for \$1.5M over an 18-month period. The cost to the three industrial participants, in the form of in-kind contributions, will equal at least 30% of the funds provided by DOE.

In April 1987 a proposal was submitted to DOE for support of a three-year project on "Development Of Technology For High-Tonnage Production Of Superplastic Steels And For Production Of Net-Shape Products." Following a number of discussions with DOE staff and a meeting at LLNL, which included the industrial participants, a revised version of this proposal was prepared. In the revised proposal, the original three-year period was divided into two 18-month phases, with the required industry in-kind contributions committed for Phase I. Three main objectives were to be addressed in Phase I: (1) establish the composition and processing steps for the commercial production of UHCS with the development of the prerequisite microstructures; (2) produce and process the UHCS under industrial-production conditions; and (3) demonstrate the die-filling capability of the UHCS by superplastically forming a complex net-shape part. The project will also evaluate the production of UHCS components under conventional-forming methods (rolling and forging) as well as under SP-forming conditions. Emphasizing the theme of this phase, the title of the proposal was modified to "Development Of Superplastic-Steel Processing." A techno-economic analysis for the production, processing, and use of these steels was to be prepared under Phase I. Authorization for Phase I was given on March 8, 1988 with the effort at LLNL being initiated a week later.

An outline of the tasks to be performed in Phase II was presented in the revised proposal. In Phase II, we will attempt to optimize the UHCS composition in order to maximize the economics in the production, processing, fabrication, and use of SP UHCS. We will also attempt to extend the technology in the use of UHCS to other processes such as gas-pressure forming, extrusion, deep drawing, solid-state roll bonding, and solid-state pressure bonding. Continuation of the project into Phase II will require additional commitments from industry.

Personnel associated with the project at each of the participating organizations are listed below.

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Alfred Goldberg (Project Leader), Oleg D. Sherby (Consultant, overall involvement), Michael J. Strum (overall involvement, especially transformation studies and process development), Richard R. Vandervoort (compression studies), Kerry L. Cadwell (strain-rate-change studies and general support), Chol Syn (miscellaneous technical support), Mary M. LeBlanc (testing), Kevin P. Conners (testing), Edwin M. Sedillo (processing and heat treating), Robert P. Kershaw (metallography), Richard J. Gross (metallography and heat treating), and Pat I. Heth (technology transfer).

In this report we present a summary of the work performed and the results obtained covering the period from the start of the project up to March 15, 1989. It describes mainly the progress made in addressing the first objective listed for Phase I.

**2. COMMENTS ON THE SELECTION OF POTENTIALLY SUCCESSFUL
UHCS COMPOSITIONS**

The success of the project depends on the selection of a UHCS alloy composition that can readily be continuously (strand) cast and economically processed to produce the required microstructure of ultrafine ferrite grains and spheroidal carbides. The composition must also enable achieving economical, viable SP forming rates. These could be realized by raising the ferrite-stability temperature range and decreasing the grain size. Both silicon and aluminum raise the ferrite-stability temperature range. In addition to forming under SP conditions, forming under conventional methods must also be considered in evaluating the economic benefits to be derived in the use of UHCS alloys. For example, the unusually ultrafine microstructures in the processed UHCS alloys would provide for improved forming characteristics and surface finish, as well as result in better mechanical properties than those obtained in current commercial-grade steels, both steels being formed by conventional methods.

An intermediate stage in arriving at the final SP microstructure in UHCS alloys is the development of fine pearlite devoid of any proeutectoid-carbide network. Such networks, which are present in as-cast plain UHCS and UHCS-Si materials, are readily eliminated in small section sizes by hot and warm working (HWW) down through the proeutectoid-carbide-precipitation range. The self-heating during the deformation of thick sections, however, can lead to excessively slow cooling rates, which may result in carbide-network precipitation.

In a significant recent discovery it was determined that carbide networks can be eliminated in UHCS by alloying with aluminum. Work at Stanford University had revealed that no proeutectoid carbides could be detected in a UHCS material containing a sufficient amount of aluminum when furnace-cooled from 1200°C (2192°F). Work at Caterpillar on a series of UHCS-Al materials further verified that the presence of aluminum could greatly reduce or even eliminate the presence of carbide networks. Based on these observations a decision was made to work with UHCS-Al materials.

At LLNL, five UHCS-Al materials were evaluated with various nominal aluminum contents. The five materials were processed from 227-kg (500-lb.) ingots. A number of criteria were selected to establish the optimum aluminum content within the evaluated range. The criteria are based on the ability to develop the required microstructures and on the resulting SP properties. The investigations to-date show that the required microstructures and SP behavior are attainable for UHCS within the range of aluminum content evaluated.

Both an increase in the amount of hot working (HW) and a decrease in the HW temperature tend to favor the formation of proeutectoid carbide networks. Either very slow cooling, which anneals out the deformation-induced defects (potential nuclei), or very rapid cooling, which retards nucleation of the carbides, however, may reverse this tendency.

For a given HW condition the degree to which carbide networks form decreases with an increase in aluminum content. Networks are generally difficult to resolve with the light microscope. In the alloys falling within the lower end of aluminum additions the networks are nearly continuous and are limited to 0.3 μm in thickness. In the alloys falling within the higher end of aluminum additions only a small amount of discontinuous networks are present and are limited in thickness to 0.2 μm . Discontinuous and thin networks are readily eliminated by appropriate DET or DETWAD steps. Similar trends were observed by Caterpillar in their studies of UHCS containing different aluminum additions.

To-date, the maximum SP strain rate that we have achieved for the UHCS-Al alloys is 10^{-2} per second (60% per minute) at a flow stress of 131 MPa (19 ksi). At a strain rate of 10^{-3} per second the flow stress for this sample was 51 Mpa (7.4 ksi). An increase in this strain rate as well as a decrease in this stress level can be realized by further refinement and stabilization of the microstructure and/or by increasing the allowable forming temperature through increasing the upper temperature limit for ferrite stability. To achieve these objectives, the influence of various alloying additions to our basic UHCS composition will be investigated. As a first trial, hot-rolled billets of four new UHCS compositions, containing additions of aluminum, chromium, and molybdenum, have been received and phase-stability studies on these alloys have been initiated. In addition to these four new alloys, we have also received hot-rolled billets of our standard UHCS-Al and of a UHCS-Al-Si material, the latter containing a silicon content expected in a continuously cast commercial heat. These six materials were cast as 136-kg (300-lb.) ingots.

The costs for fabricating, joining, machining, and assembling of components are important factors to be considered in the design of steel structures. Taking advantage of having SP forming capabilities in a high-carbon steel could result in large savings by eliminating or greatly reducing the need for these operations. Evaluations of various structural components which could be modified so as to provide substantial savings through SP forming are currently being made at Caterpillar.

3. UHCS-AL PHASE DIAGRAM

In order to be able to evaluate and establish the thermomechanical processing parameters for the UHCS-Al alloys, it was necessary to evaluate the influence of aluminum on modifying the Fe-C phase diagram. This was done for UHCS-Al materials with three different carbon contents all containing Cr and Mn. Heating-and-quenching experiments, involving hardness measurements and microstructural observations, were used to establish the A_1 and A_{cm} boundaries. Additional information, including data for estimating solidus and liquidus temperatures was obtained from heating-and-cooling (temperature-versus-time) curves, from DTA measurements, and from dilatometric curves. The A_1 temperatures for the five UHCS-Al compositions used for the processing studies were established.

The heating-and-quenching experiments showed that the microstructure and hardness for a given quenching temperature are path dependent. Differences in hardness values and microstructure obtained between cooling down and heating up to the quenching temperature are attributed to corresponding differences in the amount of carbon dissolved in austenite. These experiments

verified that the presence of aluminum greatly retards the precipitation of proeutectoid carbides from the austenite in the absence of associated deformation.

A modified Fe-C phase diagram is proposed for our standard UHCS-Al. Aluminum is shown to slightly raise the liquidus and solidus temperatures, increase the maximum solubility of carbon in austenite, increase the A_1 temperature, and introduce a three-phase region consisting of ferrite, austenite, and carbide. These changes could favor UHCS processing and/or superplastic forming.

A comparison of the influence of aluminum and silicon on the Fe-C phase diagram was made. These elements behave similarly, in that both aluminum and silicon raise the A_1 transformation temperature, an important requirement for enhancing SP behavior. Studies on a UHCS-3Si material revealed, however, that silicon narrows the gamma field i.e., the solidus line is lowered, the A_{cm} line is raised, and the maximum solubility of carbon in austenite is decreased. On the positive side, silicon widens the temperature range where the three phases, ferrite, austenite, and carbide, coexist. These results suggest that the combined addition of aluminum and silicon may be a potentially useful approach to enhancing SP properties in UHCS alloys.

The equilibrium three-phase, ferrite-plus-austenite-plus-carbide, temperature range for each of the new UHCS materials is being identified by heating-and-quenching experiments and by dilatometric analysis. This three-phase region is bounded by the equilibrium two-phase, austenite-plus-carbide and ferrite-plus-carbide regions at the upper- and lower-temperature boundaries, respectively. The corresponding A_{cm} temperatures for each alloy can readily be obtained from the dilatometric curves.

4. CONDITIONING OF AUSTENITE

A number of studies were performed to determine the influence of the finishing temperature during hot working on the austenite grain size, on the resulting pearlitic microstructure, and on the ferritic microstructures obtained after subsequent processing. A wide range of austenite grain sizes were obtained by first heating to 1100°C (2012°F) and subsequently rolling to a constant reduction at a series of temperatures from 1100°C down to low in the austenite-plus-carbide region. Hot rolling refines the austenite grain size as well as improving microstructural uniformity, largely because of concurrent homogenization; however, neither the extent of such deformation nor the austenite grain size produced measurable variations of either the pearlite colony size or pearlite fineness. In addition, the austenite grain size did not appear to affect the final ferrite grain size. The ferrite grain size was shown to be influenced primarily by the deformation introduced during DETWAD processing, and by any additional warm working. Working the UHCS material in certain

temperature ranges of austenite, however, could result in some coarse proeutectoid carbides appearing with the pearlitic microstructure.

5. DET PROCESSING OF UHCS-AL

Conventional DET processing consists of air cooling from low in the austenite-temperature range. This procedure proved to be unsuccessful for UHCS-Al materials. Its failure is explained in terms of the transformation kinetics. With additions of aluminum to UHCS the transformation of austenite becomes sufficiently sluggish such that air cooling results in extensive austenite undercooling, and a lamellar structure forms rather than a DET structure.

Various thermal studies were aimed at spheroidizing the lamellar pearlite remaining after a conventional DET step; this included cycling through the A_1 , long-time exposures below A_1 , and cold working before the thermal exposure. None of these proved to be successful in achieving and maintaining the required ultrafine two-phase microstructure. Warm working below A_1 did accelerate the spheroidization process but it proved to be only partially successful.

Isothermal transformation studies revealed that the divorced-eutectoid transformation was strongly favored over the lamellar-eutectoid transformation at temperatures near the A_1 . Virtually complete spheroidization can be obtained by transforming at temperatures down to about 50°C (90°F) below A_1 for the UHCS-Al materials. At lower temperatures the pearlitic structure becomes increasingly more prevalent as the transformation temperature is further decreased. The transformation kinetics became increasingly more sluggish with an increase in aluminum content.

The slow eutectoid-transformation kinetics for UHCS-Al should provide a relatively wide window of temperature and time for obtaining a uniform through-thickness spheroidized microstructure using a modified DET process involving isothermal transformation. The sluggish kinetics would also facilitate controlling the self-heating during the DETWAD process by allowing cooling to take place from above A_1 to below A_1 and holding at this temperature prior to initiating the deformation. The slow kinetics also offer the opportunity for various combined DET and DETWAD steps to optimize the carbide size, carbide morphology, and the ferrite grain size. A study has been initiated to establish the isothermal TTT curves for the standard UHCS-Al material. A TTT test matrix was completed, sufficient to provide guidance for evaluating various processing steps.

6. UHCS-AL DETWAD PROCESSING

Laboratory warm-working studies were performed using 12.7-mm (0.5-in.)-square and 19-mm (0.75-in.)-square bars cut from the hot-rolled material. Conventional DETWAD processing, involving soaking at about 30°C (54°F) above the A₁ followed by additional reduction by rolling, produced fully spheroidized microstructures for all UHCS-Al materials irrespective of the degree of prior hot working. In all cases any carbide networks that may have been present in the hot-worked structures were completely eliminated.

A number of slabs of our standard UHCS-Al material, measuring 76 mm (3 in.) thick, were subjected to two parallel series of warm rolling, with total reductions ranging from 3:1 to 23:1 in thickness. Two series were rolled from two different soaking temperatures. The processing resulted in a mixed structure of spheroidal carbides and lamellar pearlite in a ferrite matrix. Various amounts of reductions were evaluated; the fraction of lamellar pearlite decreased with an increase in the amount of reduction. Large reductions also produced the finest ferrite grains. Much of the lamellar carbides could be spheroidized by a subsequent subcritical anneal.

DETWAD studies, as well as hot-working studies, were also performed by hammer forging billet sections of UHCS containing various amounts of aluminum. The billets were 75 mm (3 in.) long by 152 mm (6 in.) square. All forgings showed good surfaces with no sign of surface cracks. The hot-forging gave 70-mm (2.75-in.)-square bars; the DETWAD forging gave 57-mm (2.25-in.)-square bars. The hot-forged bars showed microstructures similar to those obtained after hot rolling. DETWAD-forged bars showed non-uniform through-thickness microstructures, in that much less spheroidization occurred at the center than at the surface of each bar. The degree of spheroidization also increased with aluminum content. Spheroidization was about 50% complete in the alloy containing the lowest aluminum content and 100% complete in the higher aluminum alloys up to about 6.35 mm (0.25 in.) below the surface. The non-uniform deformation associated with hammer forging (light blows were used in order to minimize self heating) may not be conducive for the conventional DETWAD process. The hammer forgings were performed at a commercial forging shop.

In the laboratory-processing experiments at LLNL, we were able to take advantage of the information obtained from our TTT studies. Thermocouples were intrinsically welded to the part being processed. The temperature-time window revealed by the TTT studies allowed us to initially soak above the A₁ temperature and then DETWAD process the UHCS materials at sub-critical temperatures (below A₁), with the material initially remaining in the austenite phase. Processing at the lower temperatures minimized the problems associated with self heating. Isothermal

transformation prior to the final warm-deformation steps also avoided self-heating problems and resulted in uniform through-thickness microstructures in thick sections. The DETWAD processing parameters were first evaluated through rolling studies. These studies served as a guide for subsequent press-forging experiments aimed at producing sections large enough to be used for evaluating their die-filling characteristics under SP forming conditions.

A number of small slabs, approximately 40 mm (1.6 in.) thick and about 100 mm by 125 mm (4 in by 5 in.) in area were obtained by press forging. The first few slabs were sectioned to provide samples for microstructural evaluation and for strain-rate-change (SRC) studies. Following several modifications in our DETWAD processing a number of slabs were produced having through-thickness, uniform, SP microstructures. SRC tests on corresponding tensile samples gave high values of the strain-rate exponent, m (between 0.4 and 0.5), indicative of SP behavior. Disks, measuring 77.7 mm (3.06 in.) dia. by 33.0 mm (1.30 in.) thick were machined from these slabs. The disks will be superplastically formed into a complex shape and evaluated for their die-filling capability.

At Ladish, a 227-kg (500-lb.) ingot was processed to develop SP microstructures. The ingot was first hot forged, then sectioned into two billets; one billet was subjected to a DET process, the second to a DETWAD forging process. Both billets were then warm forged to further refine the ferrite-grain size. A section of each processed billet was sent to LLNL for microstructural and superplasticity evaluations. The remainder of each billet was cut into samples by Ladish to obtain compression data in the warm-working-temperature region. Based on evaluations made at LLNL, the decision was made to use only material from the DETWAD-processed billet for the die-filling studies. Two disks were machined from the corresponding section that was received from Ladish, and these will be superplastically formed together with the LLNL disks.

7. DECARBURIZATION STUDIES IN UHCS-AL

In our heat-treatment studies, which are being performed either in air or in an argon/air mixture, we noted that some decarburization had occurred. A controlled study showed that the extent of such decarburization increased with an increase in aluminum content. This is in agreement with the observation reported in the literature that the activity of carbon in iron increases with increasing aluminum additions up to some maximum aluminum content. The amount of such decarburization, however, is much less below A_1 than it is above the A_1 .

The compositions of the four new UHCS alloys being evaluated, in addition to being selected to improve the economics for SP forming, were also based on minimizing decarburization which may

occur during processing. The relative extent of decarburization, below and above the A_1 temperatures, will be examined for these four new alloys, as well as for the newly cast standard UHCS-Al and the corresponding UHCS-Al-Si material.

8. SUPERPLASTICITY STUDIES

The size, shape, and distribution of the carbide particles, the ferrite grain size, the amount of residual pearlite, and the through-thickness uniformity were microstructural criteria used in judging the potential for SP properties. Direct evidence for the presence of superplasticity was obtained through SRC studies, whereby a single sample is subjected to a sequence of strain rates ranging from 2×10^{-5} to 3×10^{-2} per second. Measuring the flow stress at each strain rate permits the determination of the strain-rate-sensitivity exponent, m , its value being a measure of the potential for superplastic behavior. The value of m is calculated from the relationship:

$$\sigma = k\dot{\epsilon}^m$$

where k = material constant
 $\dot{\epsilon}$ = strain rate
 σ = flow stress, and
 m = strain-rate-sensitivity exponent

The value of m was obtained from the slope of a plot of $\log \sigma$ versus $\log \dot{\epsilon}$. Materials exhibiting values of $m \geq 0.4$ were considered as behaving superplastically. We selected an SRC test temperature of 750°C as a standard. Additional tests on some of the processed materials were performed at both below and above 750°C , with some tests being in the three-phase (ferrite+carbide+austenite) region. The majority of SRC tests were performed on the standard UHCS-Al alloy. Samples of other compositions were also tested.

In our studies aimed on selecting an acceptable aluminum addition for both SP properties and strand casting, SRC tests were performed on UHCS containing four different aluminum contents. Samples for SRC tests were machined from materials that had been DETWAD processed by rolling 19-mm (0.75-in.) thick coupons to a small reduction in a single pass. Completely spheroidized microstructures were obtained. The "small-strain" series of rolled materials (reductions of 4:1 or less) exhibited significantly lower strain-rate-sensitivity-exponent values, m , than did the "large-strain" rolled standard UHCS-Al material (reduction of 16:1) discussed in Section 6. This difference was surprising since the "large-strain" rolled material contained about 10% pearlite whereas the "small-strain" rolled materials the carbides contained no pearlite, being virtually

completely spheroidized. For the latter materials the "m" values increased with an increase in the aluminum content. The SEM studies of tested samples revealed the presence of clusters of etch-resistant regions (using a nital etch) in the "small-strain" materials. We believe that these clusters are associated with the presence of low-angle ferrite grain boundaries with an effective grain size of 4 to 6 μm , the actual ferrite grain size being about 2 μm . By contrast, the "large-strain" material revealed well-defined (high-angle) grain boundaries with a ferrite grain size of as small as 0.7 μm . The low "m" values are thus largely attributed to the presence of low-angle boundaries.

The above comparisons were made on materials having different hot-working histories prior to DETWAD processing. In addition, the soaking temperatures for the "large strain" and "small strain" processing were different relative to the A₁ temperatures. Thicknesses prior to DETWAD processing were also different, being 76 mm (3 in.) and 19 mm (0.75 in.), respectively. The importance of the amount of reduction during DETWAD processing on microstructure and SP properties, therefore, was re-evaluated using material having the same history prior to DETWAD processing.

Three slabs of our standard UHCS-Al material, which were obtained from a hot-rolled billet measuring 152 by 152 mm (6 by 6 in.) in cross section, were DETWAD processed to give three different total reductions in thickness. The slabs were austenitized at a temperature of 30°C above the A₁ for this alloy. The number of rolling passes used were 8, 15, and 19, respectively. In the previous DETWAD-processing studies with the standard UHCS-Al alloy we noted that processing at too low a temperature resulted in some residual pearlite, the amount decreasing with an increase in reduction. In the present studies we noted that too high a processing temperature could also result in some new pearlite, due to self heating, especially in the center of the rolled plate. Whereas regions near the surfaces and edges of a DETWAD-processed plate showed complete absence of pearlite, often some pearlite would be present at the center of the plate, independent of the amount of rolling reduction. Consistent with these observations, the "m" values obtained for SRC samples machined from the surface or edge regions were frequently higher than those values obtained for samples machined from the center region of the same rolled slab. Both sets of "m" values were independent of the amount of reduction; however, the maximum strain rate in the SP range increased with an increase in the amount of rolling reduction for both edge (or surface) and center regions.

The SEM studies showed that no significant differences in microstructure resulted from differences in the amount of reduction. Samples taken from near the surface and edge regions all had fine ferrite grain sizes with the average grain size being slightly less for the material subjected to the highest reduction. Of special significance, however, is that the difficult-to-etch regions were

virtually absent in these samples, which experienced a relatively large number of rolling passes. By contrast, such regions were quite prominent in the series of materials that were DETWAD-processed with a single-pass reduction described earlier in this section. Thus, the discrepancy between the earlier and more recent observations on the importance of the amount of reduction during DETWAD-processing is attributed to differences in the extent to which low-angle ferrite grain boundaries are present, since such boundaries adversely affect the SP properties. Our most recent investigations have led to processing methods which can eliminate, or at least greatly minimize, both through-thickness non-uniformity and the presence of large etch-resistant regions. The new processing methods have led to average ferrite-grain sizes in the 1-to 2- μm range. Some small amount of growth of both carbide particles and ferrite grains is obtained during an SRC test.

A brief study was made on the influence of silicon on the superplastic behavior of UHCS-Al. As mentioned in Section 3, it was thought that a combination of Al and Si could enhance the superplastic properties of UHCS. This is because the temperature range of ferrite stability is increased and the three-phase (ferrite-plus-carbide-plus-austenite) region is widened. Two UHCS-Al-Si castings were prepared with two different silicon additions. Although it was possible to develop a superplastic microstructure by DETWAD rolling, surface cracking was commonly observed during such warm rolling. Furthermore, graphitization occurred in the material containing the higher silicon addition. It was decided, therefore, to discontinue studies on this material. As pointed out in Section 2 of this report, we are newly directing a considerable amount of our effort to examining other alloying additions to our basic UHCS composition in order to raise the temperature range for ferrite stability and thereby achieve an increase in SP strain rates and a decrease in SP flow stresses.

9. ROOM TEMPERATURE MECHANICAL PROPERTY STUDIES

Exploratory mechanical-property studies at room temperature revealed that the standard UHCS-Al alloy had good cold-rolling characteristics and exhibited high hardness with good bend ductility. The material can be cold rolled, without annealing, to 50% reduction in the fully pearlitic condition and to over 60% reduction in the fully spheroidized-and-annealed condition. The DETWAD-processed UHCS-Al alloy had hardness values on the order of RC 35 and bend ductilities in the range of 10 to 20% equivalent tensile strain at the start of cracking. Tensile data were obtained for three conditions of the DETWAD-processed standard UHCS-Al alloy: (1) annealed at 650°C, (2) annealed plus 50% cold-rolled, and (3) reheated into austenite-plus-carbide region and air cooled to form pearlite. Yield strengths were of the order of 860, 1,170 and 755 MPa (125, 170, and 110 ksi), respectively, for the three conditions. The corresponding ultimate tensile strengths were about 900, 1,185, and 1,100 MPa (130, 172, and 160 ksi) with elongations of 21, 12, and 12%,

respectively. We tested the 650°C-annealed condition in both transverse and rolling directions with identical results being obtained.

10. CONTINUOUS CASTING EFFORTS

Based on the results described above, on discussions held at the Technical Meeting of August 17, 1988, and on subsequent discussions held between LLNL and the industrial partners the decision was made to cast an Fe-1.35C-Al-Si-Cr-Mn alloy (with nominal amounts of impurities). North Star Steel evaluated the various factors to be considered in producing this first heat. They also reviewed work done by others in the production of aluminum-containing steels. The first attempt at continuous casting resulted in a breakout of the shell in the mold on startup. A number of possibilities were considered for the failure, amongst which were cooling problems associated with formation of an oxide film, excessive slag entrapment, poor hot ductility, poor hot strength, and the presence of a low-melting-temperature phase. The degree of mold taper required to accommodate the contraction characteristics of the UHCS during solidification and cooling was also considered. To aid in resolving the source of the problem, DTA and dilatometric studies were initiated.

The DTA analyses were performed on a North Star Steel 1090 steel and on material left in the tundish. The analysis of this material was well within limits of the desired composition. Dilatometry was performed on a series of plain-carbon steels (1010, 1018, 1045, and 1090) and on a number of UHCS alloys in which the effects of Al, Cr, Mn, Si, C, individually and combined, on the coefficient of expansion were evaluated. The DTA samples permitted an estimate of the liquidus and solidus temperatures. Combined with other data, possible solutions to the casting difficulties were formulated.

The decision was made to attempt a second heat after making a number of modifications that would either eliminate or minimize the previous problems. In addition, at startup of strand casting, two of the four parallel strands would be set to run at different speeds. Finally, the composition of the UHCS-Al alloy was modified, but only in the elimination of silicon.

The second strand-casting attempt again resulted in a shell breakout. The shell showed no evidence of slag entrapment. It was approximately 10 mm (0.4 in.) thick, typical of successful castings. Examination of the shell suggested the basic causes for the two failures. Before proceeding with a third full-scale heat, a laboratory-size heat will be made on an experimental continuous caster. Although this caster has a straight mold, we should be able to assess the

behavior of the UHCS-Al alloy during continuous casting, divorced from the mold-taper problem, as well as being able to examine the as-cast structure.

11. MISCELLANEOUS

Meetings

Three technical exchange meetings, attended by representatives of Caterpillar, Ladish, North Star Steel, and LLNL were held as follows:

May 18-19, 1988 at Ladish, Cudahy, WI

August 17, 1988 at North Star Steel, Monroe, MI

December 6-7, 1988 at LLNL, Livermore, CA

The next meeting is scheduled for April 10-11, 1989 at Caterpillar, Peoria, IL.

Financial

Estimated expenditures to March 30, 1989 are as follows:

LLNL - \$823,000 (includes \$28,000 obligations)

Caterpillar - \$140,000

Ladish - \$54,000

North Star Steel - \$160,000

Total Industrial contribution of \$354,000 is equal to 43% of LLNL costs.

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