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DEVELOPMENT OF SUPERPLASTIC STEEL PROCESSING

Final Report

By
A. Goldberg et al.

April 1995

Work Performed Under Contract No. W-7405-48

For
**U.S. Department of Energy
Office of Industrial Technologies
Washington, D.C.**

By
**Lawrence Livermore National Laboratory
Livermore, California**

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EXECUTIVE SUMMARY

This document is a final report for the LLNL-Industrial Project "Development Of Superplastic Steel Processing" initiated under the Department Of Energy Steel Initiative Plan. The Project extended over three separate periods referred to as Phase I, Phase IIA, and Phase IIB. Phase I was initiated in March, 1988 between the Lawrence Livermore National Laboratory, Caterpillar, Inc., North Star Steel Company, and Ladish Co. Inc. Phase IIA started in June, 1990 and was completed at LLNL in June, 1991. Phase IIB was initiated in October, 1991 and was scheduled for terminating in March, 1994. Efforts at both Caterpillar and North Star Steel are expected to continue beyond this date. Caterpillar and North Star Steel supported the Project through the end of Phase IIB, whereas Ladish discontinued their support at the end of their commitment for Phase IIA. A total sum of \$4,026,000 was funded by the DOE for the three Phases, with in-kind contributions by the industrial participants exceeding the required 30 percent of the funds provided by DOE.

The principal objective of the project was to provide the basis for producing, processing, and forming UHCS materials on a commercial scale. This was demonstrated for a basic composition of ultrahigh carbon steel (UHCS) containing 1.6% Al and 1.35% C. Based upon these results, business plans were developed with the assistance of outside consultants which point out the areas of potential commercialization as well as the requirements to enhance the commercialization of UHCS materials. A preliminary economic analysis in Phase I pointed out the need for further improvements in superplastic (SP) forming behavior in order to make SP forming competitive with more conventional technologies. Therefore, a significant portion of the effort in this project was directed at improving the combination of flow stress and forming rates in UHCS alloys. The tasks in Phase I and Phase II were subsequently largely redirected toward optimizing the SP properties through modification of UHCS compositions and processing methods. These efforts resulted in the development of a series of UHCS alloys and processing methods, the selection of which is expected to depend upon the specific requirements of the commercial application. A patent titled, "An Improved Transformation Process for Production of Ultrahigh Carbon Steels and New Alloys" which includes both advanced processing methods and new UHCS alloys, is pending final patent office action. In addition to improvements in flow stress by raising the temperature window for SP forming, and improvements in SP forming rates through improved microstructural refinement and stabilization, useful ancillary properties were also developed in these materials. These include compositions with improvements in mechanical properties, wear resistance, and oxidation resistance at elevated temperatures. It was also recognized that the economics of SP forming are improved by the existence or incorporation of design elements in candidate components which utilize the unique advantages of near net shape SP forming. To help evaluate these capabilities of near net shape forming, modeling

studies were performed by LLNL and Caterpillar, the results of which provide guidance in creating die designs and reduction schedules for SP forming.

In Phase I, aluminum additions to UHCSs were established which provide as-cast microstructures amenable to conversion to SP microstructures and which have been demonstrated as capable of being continuously cast on a pilot plant horizontal caster. Aluminum concentrations were identified which greatly reduce the tendency for the formation of brittle carbide networks, a problem which has largely prevented more widespread commercial application of high carbon steels containing proeutectoid carbides. Conventional hot rolling treatments were demonstrated as suitable for ingot breakdown of the Al-modified alloys. Increases in aluminum concentrations also provided an increase in the peak SP forming temperature by increasing the ferrite transformation temperature (A_1). This temperature dependence was experimentally evaluated and improvements in maximum SP forming rates were achieved through increases in SP forming temperature. Aluminum additions, however, also introduced changes in the SP processing response and could introduce problems in the continuous casting (CC) of this material. Taking this into consideration, an optimized base aluminum content was established. To support this decision, characterization of aluminum effects on the Fe-C phase diagram and on the austenite transformation kinetics were completed. These results were used to successfully develop and demonstrate appropriate processing schedules for the production of uniform SP microstructures in thick section bar and plate in these alloys.

Attempts at continuous (strand) casting two 130-ton heats of a low-aluminum UHCS during Phase I proved to be unsuccessful, although problems were not encountered on a 500 lb (227 kg) pilot-plant heat of the same material. The attempts at casting the two large heats were made on a curved-mold, curved-strand caster, whereas the 500-pound heat was made on a straight-mold, vertical-type caster. The processing and SP properties of this alloy were extensively investigated during Phase I. The development materials were obtained from 500 lb (227 kg) ingot castings similar in cross-section to North Star Steel strand castings. In addition, studies were performed to provide physical and mechanical property data to North Star Steel for strand casting development.

Forgings of subscale commercial parts were superplastically formed at Pratt & Whitney and Ladish for die-filling studies. These tests provided valuable information on die-filling capabilities of the UHCSs and strain-assisted microstructural changes, although quantitative analysis of the effects of the parameters controlling microstructural changes were better measured from mechanical test specimens of uniform cross section.

Based on an analysis made by Ladish on SP forming of a Caterpillar part, specific goals were set for increased SP strain rates and decreased flow stresses. These goals were based on the perceived need for hot-die vs. isothermal forging

practices, which require relatively rapid strain rates and flow stresses compatible with tool steel dies. Theory and extrapolation of existing data indicated that the desired SP strain rate should be achievable with ferrite grain sizes of the order of 0.5 μm , a factor of about four less than that achievable in Phase I.

The need to solve the strand casting problems, the need to raise the SP strain rates and lower the forming stresses, the need to integrate the die-filling experiments with analytical modeling studies, the need for providing ambient-temperature properties, and, finally, the requirement of demonstrating the techno-economic advantages by SP forming a commercial part set the framework for Phase IIA, which continued into Phase IIB. In this document, in reporting on Phase II, distinctions are generally not made between Phases IIA and IIB. In addition, because of the proprietary nature of the Project, many of the precise details on processing and material properties are omitted. These details have been presented at the Technical Exchange Meetings.

The main effort at LLNL in Phase II was directed towards increasing the fineness of the microstructure and retarding the as-processed dispersoid size and grain size from growing during SP deformation. A number of approaches were pursued, including: 1) increasing the concentration of strong carbide formers, 2) developing improved processing procedures, 3) introducing ultrafine dispersions using powders, 4) evaluating spray casting, and 5) introducing nitrogen to form an ultrafine dispersion of highly stable aluminum nitrides (AlN). Some of these approaches led to significant decreases in grain sizes. The problem of carbide growth and resulting grain growth during SP deformation, however, still existed. It now appears that the SP strain rates achieved in Phase II may be the upper limits for UHCSs in which the grain-size stability is based on carbide pinning of grain boundaries and in which the alloys are formed by current methods using either ingot or continuous casting. Although the additions of strong carbide formers appeared promising, difficulties were encountered in eliminating the coarse carbides originating in the ingot castings and this approach was discontinued. By increasing homogeneity through the use of magnetic stirring and increasing solidification and cooling rates through strand casting, this problem should be greatly reduced. The introduction of ultrafine dispersions by alternate methods was explored through formation of alloy carbides from powders and through formation of highly stable nitrides by introducing nitrogen into the melt. The results of these studies indicate that numerous possibilities exist for both particle-size and grain-size stabilization. Furthermore, using the AlN approach could eliminate the need for carbides. A sufficient reduction in the carbon content could also allow for an increase in the fracture toughness of these SP steels.

A second important effort at LLNL was the development of a test facility whereby the flow properties and die-filling capability of a material could be integrated with numerical analysis. This facility, in addition to verifying the analytical simulations, was also used to evaluate different die lubricants and the

response to different strain rates and different microstructures (non-SP versus SP). A simple screening facility was also developed that was valuable for rapid screening of new alloys and processing methods. A third important effort was that of providing input for continuous casting of UHCSs. Included were measurements of thermal coefficients of expansion, solid-state transformation temperatures, solidus and liquidus temperatures, and hot-hardness data. Some exploratory studies were made on the effect of microstructural modifications on fracture toughness as affected by alloying, heat treatment, and/or processing. Exploratory studies were also performed on joining UHCSs using diffusion bonding and inertia friction welding.

Continuous casting trials by North Star Steel in Phase II were made on horizontal casters at First Mississippi Corporation. The first attempts were with a small caster using 1000-pound heats. Successful casts were made with a low-aluminum UHCS. Attempts with a high-aluminum composition were unsuccessful and further casting trials with this alloy were postponed until appropriate corrective measures were made. The success with the 1000-pound heat of the low-aluminum UHCS was followed by two unsuccessful attempts of continuous casting 40-ton heats of the same alloy. The 40-ton heats were then successfully cast into a number of large diameter ingots. The structure of these ingots was deemed to be unacceptable for further processing into demonstration components by Caterpillar due to excessive segregation and carbide agglomeration. These problems are not expected to be characteristic of strand castings, which are much smaller in diameter and are magnetically stirred during cooling. Before additional attempts of continuous casting were to be made, North Star Steel agreed to consult with casting experts, who could help in identifying the cause for the failures and in recommending corrective measures.

During Phase IIA, Ladish was primarily involved with the die-filling studies on subscale parts of commercial components. This involved both SP forging and interacting with LLNL on analyzing the superplastically forged parts. Although their involvement on the Project officially terminated at the end of Phase IIA, that effort was not completed until after the start of Phase IIB. In Phase IIB, LLNL also consulted with Ladish on the design of the die-filling test facility to be built at LLNL.

Caterpillar's efforts throughout Phase II were directed primarily to obtaining engineering property data. In the latter part of Phase II, Caterpillar focused on supporting and coordinating the SP forming of a demonstration part to be used in evaluating the techno-economic potentials for UHCSs. This involved the acquisition, processing, and machining of material for forging preforms and the interaction with Pratt & Whitney on modeling, verification forging runs, and final production of the demonstration part.

Two consulting firms, one identified by LLNL and, later, a second identified by Caterpillar, created business plans on the commercialization of UHCSs. In both

analyses, numerous examples were given of potential uses for UHCSs in sheet, plate, and/or bulk forms which expand upon the applications considered by the industrial participants in this Project.

1. INTRODUCTION

1.1 Project Chronological Background

Extensive studies at Stanford University under the direction of Professor Oleg D. Sherby demonstrated that, by appropriate thermomechanical processing, ultrahigh-carbon steels (UHCS) could be made to behave in a superplastic fashion at moderately elevated temperatures. Furthermore, the ultrafine microstructure developed by this processing resulted in the enhancement of ambient-temperature tensile strength and ductility combinations. Superplastic forming of net- or near-net-shape commercial products has been used for titanium-, nickel-, and aluminum-base alloys and most recently for duplex stainless steels. The successful economical production of these products demonstrated the potential for large savings in machining, scrap-handling, joining, and material costs by using SP forming. Also, SP forming may provide a means of producing complex configurations that could not readily be attainable by conventional fabrication techniques. The success with current commercial SP alloys and the encouraging observations made by Sherby on UHCSs suggested that, with a sufficiently expanded effort, SP forming of UHCSs on a commercial scale could be demonstrated as being economically attractive. Therefore, in response to requests for proposals by the Department Of Energy (DOE), which was responsible for managing the Steel Initiative Plan (renamed Metals Initiative Plan), a proposal was submitted by the Lawrence Livermore National Laboratory (LLNL) in April, 1987 for a research and development project that would lead to the commercialization of UHCSs.

The proposal, which was submitted initially for a three-year project, was divided into two phases. Phase 1, which started in March, 1988, was funded at a level of \$1,500,000, with commitments of in-kind contributions by the industrial partners of at least 30 % of this sum. Phase I was completed in March, 1990. Phase II was initiated in June, 1990 following the signing of the "Intellectual Property Management Agreement" between the University of California (UC) and the DOE and the "Participation Agreement" between UC (for LLNL) and the industrial participants. Phase II was divided into two phases, designated as A and B, with funding initially provided for Phase IIA at a level of \$706,000; \$1,800,000 was later committed for Phase IIB. In-kind support of 30 % of these sums was committed by the industrial participants. Phase IIA was funded for a period of 11 months, through April, 1991. At LLNL, this was extended through June, 1991. Phase IIB was initiated in October, 1991 following the signing of the Licensing Agreement between UC and Dr. Sherby. Work on the Project at LLNL was completed in November, 1993.

The studies performed and knowledge gained during the Project were discussed in three previous reports presented to the DOE. The reports, which were prepared by Alfred Goldberg of LLNL, are referenced by the DOE under "Steel Initiative" and are identified below:

DOE/CE-0256--First Semi-Annual Progress Report On Development Of Superplastic Steel Processing, March 1989. (Covers the period March 15-September 15, 1988.)

DOE/CE-0256-1--First Annual Progress Report On Development Of Superplastic Steel Processing, September, 1989. (Covers the period March 15, 1988-March 15, 1989.)

Third Progress Report On Development Of Superplastic Steel Processing. (Covers the period March 16, 1989-March 15, 1990.)

1.2 Project Participants

Personnel associated with the project at each of the participating organizations and principal suppliers are listed in the following.

Participating Organizations.

North Star Steel Company: Jerry D. Thomas, Gordon H. Geiger, and John I. Stipancich;

Ladish Co. Inc.: (Participated only in Phases I and IIA) Arthur F. Hayes and Christopher J. Misorski;

Caterpillar Inc.: Donald H. Sherman, Gary L. Biltgen, Ken Erickson, and Egon E. Wolff

Lawrence Livermore National Laboratory: Alfred Goldberg (former Project Leader/retired), Richard L. Landingham (former Project Leader/retired), Edward N. Dalder (current Project Leader), Michael J. Strum (alloy development, thermomechanical processing, and fracture toughness), William W. Feng, Daniel J. Nikkel, and Frank H. Magness (modeling/die filling), Kerry L. Cadwell (physical, thermal, and mechanical properties), Anne J. Sunwoo (joining), Clifford W. Price (microstructural and chemical evaluations), Donald R. Lesuer (miscellaneous support), Oleg D. Sherby (consultant, Phase I and IIA only), Paul G. Curtis, Richard J. Gross, Edwin M. Sedillo, and C. Scott Preuss (technician support), and Pat I. Heth (technology transfer);

Principal Suppliers.

Homer Research Laboratory, Bethlehem Steel Corporation, Bethlehem, PA: Bruce L Bramfitt (ingot casting, ingot breakdown, and process rolling);

Coulter Steel and Forge Company, Emeryville, CA: Thomas Coulter and William Mendell (ingot breakdown and process forging);

Pratt & Whitney, Engineering Division South, West Palm Beach ,FL: Steve McLeod and Bryant H. Walker (SP forming and die filling of subscale parts);

Pratt & Whitney, Columbus, GA: Steve McLeod (modeling and SP forming of Caterpillar demonstration part);

RMI Company, Niles,OH: Gerald H. Filipisky (ingot breakdown and process forging);

MIT, Cambridge, MA: Nicholas Grant (spray casting and powders);

EG&G, Idaho Falls, ID: John Flinn (powders).

2. PROPOSED TASKS

Tasks proposed for Phase I. The principal tasks proposed for Phase I were as follows:

1. Establish an aluminum content for UHCS materials which provides as-cast microstructures amenable to conversion to superplastic (SP) microstructures and which is also capable of being continuously (strand) cast.
2. Develop the technology and demonstrate the feasibility for strand casting UHCS-Al materials.
3. Develop the processing steps to produce SP microstructures through the entire thickness of thick section bar and plate.
4. Demonstrate the die-filling capability of the processed material when SP formed into a complex part.
5. Establish the window of parameters for SP forming.
6. Obtain room temperature mechanical property data.

7. Perform a techno-economic analysis for the commercial production and industrial use of SP UHCS materials.
8. Direct studies to increase SP strain rates and decrease corresponding flow stresses. (Note: this task was introduced late in Phase I).

Tasks proposed for Phase IIA. The main tasks proposed for Phase IIA were as follows:

1. Continue evaluations of modified UHCS compositions and processing methods aimed at optimization of SP properties.
2. Select a composition for evaluating the commercialization of UHCS materials.
3. Develop the knowledge for continuous casting of UHCS materials.
4. Cast ingots of a selected UHCS by North Star Steel and obtain data to support selection of strand casting parameters.
5. Complete the die-filling studies at Ladish.
6. Increase the database on mechanical properties of UHCS materials.

Tasks proposed for Phase IIB. The main tasks proposed for Phase IIB were as follows:

1. Continue evaluations of new compositions and processing procedures in order to increase the fineness and stability of the superplastic microstructures.
2. Identify optimum low-aluminum and high-aluminum UHCS compositions for commercial SP forming.
3. Perform property measurements pertinent to continuous casting of selected low-aluminum and high-aluminum UHCSs.
4. Verify the castability of UHCSs on a horizontal continuous caster.
5. Produce a successful heat of continuous-cast billets at North Star Steel.
6. Identify the windows of critical parameters to obtain SP microstructures on commercial processing UHCSs by North Star Steel.
7. Process and prepare blanks for SP forming a viable commercial component.

8. Perform and verify modeling studies for SP forming.
9. Superplastically fabricate a commercial component selected to be used in evaluating the techno-economics of commercializing UHCS materials.
10. Measure properties of the superplastically formed component.

3. COMMERCIAL SCALE-UP OF PROCESSING METHODS FOR PRODUCING SUPERPLASTIC BAR AND PLATE

Composition and processing evaluations in UHCS-Al materials have led to the successful production of superplastic (SP) microstructures in thick sections. Compositions were established which provide an ultrafine pearlite without harmful carbide networks and processing schedules were established for the SP conversion to a microstructure consisting of fine-grained ferrite stabilized by a uniform distribution of fine spheroidal carbides. Typical UHCS-Al microstructures before and after SP conversion processing are shown in Figure 1. The development work was principally performed using 227 kg (500 lb) ingot castings with cross sections which approximate the size of commercial strand castings (6X6 or 8X8 inch). Forged SP blanks were produced by both LLNL and Ladish for use in die-filling studies and by Reactive Metals Inc. (RMI) for SP forming evaluations of a commercial component.

3.1 Elimination of carbide networks.

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, embrittle the steel and degrade the SP performance by tying up carbon in a form which is ineffective in producing a fine ferrite grain size. The proeutectoid carbide in a 1.25 C steel accounts for approximately 40% of the total available carbide. In order to eliminate formation of proeutectoid carbide networks, a hot and warm working (HWW) treatment has been traditionally used. The HWW process is effective in thin sections but was considered undesirable for commercial scale-up to thick sections due to: 1) excessively slow cooling rates due to self-heating during deformation, 2) requirements for large reductions in section which limit the amount of reductions available for SP conversion to a given SP blank size, and 3) additional processing cost and equipment requirements. In a significant discovery at Stanford University, it was determined 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 establish the acceptable composition limits for aluminum additions to UHCS materials in this Project.

At LLNL, a series of five UHCS-Al materials were evaluated with nominal aluminum contents between 0.5% and 2%. 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.

For a given hot working (HW) condition, the degree to which proeutectoid carbide networks form decreased with increases in aluminum content. Both an increase in the amount of HW and a decrease in the HW temperature tended to favor the formation of carbide networks. The data in Table I summarizes the microstructural parameters in HW UHCS-Al alloys vs. Al concentration. The large HW reduction of 9:1 produced a relatively fine austenite grain size of 12 to 13 μm in all the UHCS-Al compositions, and the resulting carbide networks were relatively thin. Network formation in the 0.5% Al material was nearly continuous while the network carbides became discontinuous and increasingly scattered with the addition of 1.5% and 2% Al. The networks in Al-containing UHCS alloys, when present, were readily eliminated by appropriate SP conversion processing steps. The absence of any network carbides in the 1% Al alloy is consistent with its low C concentration of 1.0% vs. the typical C concentration of 1.2%. Caterpillar studies on as-cast UHCS-Al with 1.25% C were in close agreement with LLNL data. The UHCS with 0.18 to 0.55% aluminum contained harmful carbide formations while the thinness of the carbide network in the 0.97 to 1.57% Al alloys was expected to result in its dispersion during hot working and heat treatment. The UHCS-1.65Al contained a thin carbide network as-cast but was free from harmful carbide formation after heat treatment. There was no evidence of any harmful carbide formation in UHCS-2.4Al in either the as-cast, forged, or heat-treated conditions.

A number of criteria were selected for evaluating the influence of aluminum content on the microstructures and properties of UHCS-Al alloys. These criteria were used to establish the optimum aluminum content within the compositional ranges of materials studied. The criteria selected for the laboratory studies are summarized in the following:

1. Metallography of UHCS-Al in various hot-worked (HW) conditions.
2. Metallography of UHCS-Al in DETWAD-processed conditions.
3. Metallography of UHCS-Al in DET-processed conditions.

4. Ability to develop the required superplastic microstructures in large as well as small sections of UHCS-Al by either current or newly developed DET and DETWAD processes.
5. The degree of decarburization resulting from heating in the austenite range required for the DET and DETWAD steps.
6. Superplastic properties of UHCS-Al as determined by strain-rate-change tests.

In addition to these criteria, input from North Star Steel based on their strand casting studies, from Ladish based on their forging studies, and from Caterpillar based on their composition studies were incorporated. The investigations show that the required microstructures and SP behavior are attainable for UHCS within the range of aluminum content evaluated. From these studies, Fe-1.35C-1.6Al-0.5Si-1.5Cr-0.5Mn was selected as the first North Star Steel composition to be strand cast.

Table I. Microstructural Evaluations for Establishing Optimal Aluminum Content in UHCS-Al Alloys with Reference to the Hot-Rolled Series Having a 9:1 Reduction in Area.

Alloy UHCS-	Analysis*		Austenite Grain Size μm	Pearlite Colony Size μm	Pearlite Spacing μm	Extent of Grain-Boundary Carbides
	Al	C				
0.5Al	0.49	1.17	13	3.0	0.11	Nearly continuous
1Al	1.18	1.01	13	3.4	0.07	No network carbides <0.1 μm thick
1.5Al	1.45	1.22	12	3.8	0.14	Discontinuous; 0.2 μm thick
2Al	1.83	1.18	13	3.1	0.10	Discontinuous, scattered; <0.2 μm thick

*Anamet analysis made on samples from hot-rolled materials.

3.2 Processing temperatures (phase diagram determination).

In order to best 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. Heat-and-quench experiments, involving hardness measurements and microstructural observations, were used to establish the A₁ 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 differential thermal analysis (DTA), and dilatometric measurements.

The influence of Al concentration on the A₁ temperatures is significant, as shown in Table II for six UHCS alloys (0 Al, 0.5 Al, 1 Al, 1.5 Al, 1.6 Al, and 2 Al). The A₁ temperature increases continuously with increasing Al content over this range. Increases in the A₁ temperature are considered desirable because they expand the superplastic range to higher temperatures, where flow stresses decrease and diffusional accommodation rates increase. Work at Stanford University [Ref. 1,2] expanded this data to 10% Al and examined the influence of Si additions. These results are plotted in Figure 2. The A₁ temperature is raised to approximately 940 °C for the 10 Al alloy, although Si was found to provide a higher rate of increase in A₁ temperature than Al. Further evaluations of Si containing alloys in this project are described later, but Al was selected as the preferred method of increasing the A₁ temperature.

Table II. A₁ Temperatures in UHCS-Al as Determined by Heating Curves.

Temperature	0Al	0.5Al	1Al	1.5Al	2Al	1.6Al
°C	727	754	765	772	782	779
°F	1341	1389	1407	1422	1440	1435

The various methods that were utilized to determine critical temperatures in the UHCS-1.6Al materials (1.25 to 1.8C) permit making a good estimate of the Fe(Al)-C phase diagram. It was determined that Al changes the Fe-C phase diagram in several important ways:

1. Aluminum raises the melting temperature of UHCS relative to unalloyed UHCS. For a UHCS-1.6Al alloy containing 1.4% C, melting begins at 1300°C (2372°F). Therefore, the HW range with UHCS-Al alloys is extended beyond those observed in unalloyed UHCS.
2. The maximum solubility of carbon in austenite is 2.3% C for a UHCS-1.6Al alloy, in contrast to 2.1% C in the case of unalloyed UHCS. This means that UHCS with 1.6% aluminum, can have as much as 35% carbides, all of which can be dissolved in austenite at sufficiently high temperatures.

3. The A_1 temperature increases with increasing aluminum additions. The temperature increase can be calculated from a master graph that illustrates this correlation.
4. The three-phase region—ferrite, austenite, and cementite—appears to extend over a narrow range of temperatures for the UHCS-1.6Al system. In contrast, silicon appears to be more effective in expanding this three-phase region.
5. The A_{cm} line appears to be at slightly lower temperatures for UHCS with 1.6Al addition than for the case of unalloyed UHCS.

These changes in the phase diagram with the addition of 1.6% Al are largely favorable with respect to improving UHCS processing and SP properties.

Changes in the Fe-C phase diagram due to Si additions were also evaluated from the literature and from experimental UHCS-Si alloys. Increases in the A_1 temperature by silicon addition have already been described, indicating the similarity in behavior with aluminum. Silicon, however, affects other portions of the Fe-C phase diagram in a way very different from aluminum. Silicon closes the gamma loop. In relation to the unalloyed Fe-C phase diagram, the solidus and liquidus lines decrease, the maximum solubility of carbon in austenite decreases, the A_{cm} line increases, and the eutectoid composition is shifted to a lower carbon content. These changes in the phase diagram due to Si additions were unfavorable due to increasing susceptibility to graphitization and to warm shortness in UHCS-Al-Si alloys, as discussed in section 6.

3.3 Processing windows (time/temperature/transformation diagram determination).

Conventional processing for the development of spheroidal carbides in UHCS alloys consisted of heating the material to a temperature which is low in the austenite-carbide range followed by air cooling. This procedure proved to be unsuccessful for UHCS-Al materials. The cause of the failure of conventional processing was explained in terms of changes in the transformation kinetics. Modifications to conventional spheroidization methods proved successful and led to a patent filing titled "An Improved Transformation Process for Production of Ultrahigh Carbon Steels and New Alloys".

Isothermal transformation studies revealed that the divorced-eutectoid transformation is strongly favored over the lamellar-eutectoid transformation at temperatures near the A_1 . Virtually complete spheroidization was obtained by transforming at temperatures down to about 50°C (90°F) below the A_1 temperature for the UHCS-Al materials. As the transformation temperature was further decreased, the pearlitic structure became increasingly more prevalent. Aluminum additions were determined to cause the kinetics for the transformation of austenite into ferrite and carbides to become increasingly

sluggish such that large undercoolings occur prior to completion of the transformation. For the Al contents of interest to the Project, modifications to conventional processing methods were required. It was demonstrated that, in practice, carbide spheroidization can be achieved without deformation by isothermal transformation, by interrupted cooling, or by maintaining cooling rates below a critical value throughout the effective transformation range. In Figure 3, a particular UHCS composition which was cooled at a rate of 3 °C/min contains largely spheroidal carbides (Fig 3a) while that cooled at a rate of 20 °C/min contains a significant fraction of lamellar carbides (Fig 3b).

The reduced rates of eutectoid-transformation for UHCS-Al alloys provide a relatively wide window of temperature and time for obtaining a uniform through-thickness spheroidized microstructures. This also facilitates controlling the self-heating during deformations in grain refinement treatments by allowing cooling to take place from above A₁ to significantly below A₁ prior to initiating the deformation. The slow kinetics also offer the opportunity for various combined thermal and mechanical steps to optimize the carbide size, carbide morphology, and the ferrite grain size. A study was undertaken 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.

3.4 Processing of thick sections.

Standard processing procedures, which produced fully spheroidized SP microstructures in warm rolling experiments at LLNL, could not be successfully transferred to larger scale section sizes and larger scale processing methods. Two examples were; 1) 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. Subcritical anneals after rolling were helpful in spheroidizing much of the residual lamellar carbides, but extended anneals were undesirable due to associated microstructural coarsening of the grain size. 2) DETWAD studies, as well as hot-working studies, were also performed by hammer forging billet sections of UHCS containing various amounts of aluminum by a commercial forge shop. 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 DETWAD forging gave 57-mm (2.25-in.)-square bars. These 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 increasing 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.

In subsequent processing studies at LLNL, the information obtained from the TTT characterization provided the basis for appropriate modifications to obtain uniform SP microstructures in thick sections. The temperature-time window revealed by the TTT studies allowed initiation of mechanical deformation by DETWAD-type processing to be postponed until after thermal stabilization at sub-critical temperatures (below A_1). The material, initially soaked at a temperature above the A_1 , remained as austenite and proeutectoid carbides prior to initiation of deformation. Processing at the lower temperatures provided a uniform microstructural condition through the thickness of the billets and minimized the problems associated with self heating. The improvements in microstructural uniformity achieved by these process modifications are illustrated in Figure 4, in which large fractions of residual pearlite remain at the center of continuously deformed billets (Fig 4a) and uniform SP microstructures are produced by process modifications (Fig 4b). An alternative processing schedule in which spheroidal carbides were produced by isothermal transformation prior to the warm-deformation steps also avoided self-heating problems and resulted in uniform through-thickness microstructures in thick sections. The appropriate warm deformation schedules were initially developed through rolling studies. These studies served as a guide for subsequent press-forgings in which SP blanks for die-filling experiments were produced.

3.5 Microstructural requirements.

The correlations between processing and microstructure and between microstructure and SP properties were developed and used as a basis for identifying critical processing parameters and processing windows. The results of this effort allowed the tailoring of processing steps to optimize the effectiveness of warm working reductions on enhancement of SP properties. Two key principles for the enhancement of SP properties, supported by our experiments were to: 1) create a fine grained ferrite with large grain boundary misorientations, and 2) create and maintain a fine carbide distribution. Combinations of microstructural characterization and stress vs. strain rate measurements of the SP properties identified that the improvement in SP properties with large warm working reductions were due to increased grain boundary misorientations. Grain boundary misorientations vs. warm working strains were physically measured by tilting experiments in the transmission electron microscope. This work identified the grain refinement mechanism as one of subgrain formation followed by continuous increases in misorientation with increasing strain, and not a recrystallization-type mechanism. Processing parameters were subsequently identified which optimize the benefit of warm reductions with respect to SP properties. Processing parameters to create and maintain fine carbide distributions were also identified. The grain size of warm

worked specimens allowed to stabilize at the SP forming temperature was found to be closely associated with the carbide size and distribution. These results verified the importance of a fine carbide distribution and shed new insight on the benefits of improving the carbide coarsening resistance. Warm working schedules were therefore developed which optimize the effective grain refinements while limiting carbide growth. The issue of optimizing the microstructure, especially with respect to grain refinements and carbide stabilization, through changes in the chemical composition of the alloys and through modified processing methods are further discussed in section 6 and the correlation of microstructure and SP properties are further discussed in both sections 4 and 6.

3.6 Processing and preparation of materials for SP forming demonstrations.

The scale-up of superplastic steel processing has been demonstrated on several levels. Much of the work was performed on ingot castings, but with section sizes typical of strand castings. Processing has been performed on commercial-like rolling equipment at Homer Research Laboratories, RMI, and North Star Steel, and on forging presses at LLNL, Coulter Steel and Forge, and Ladish. Several of the forgings were processed into SP blanks which were used for die fill demonstrations. Using this experience, Caterpillar directed commercial-like processing of SP blanks for use in SP forming a commercial component.

For initial die-fill evaluations, 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 were superplastically formed by Pratt & Whitney into a complex shape and evaluated for their die-filling capability, as described in a later section on SP forming demonstrations.

At Ladish, a 500 lb (227 kg) 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 were superplastically formed together with the LLNL disks.

Processing schedules were formulated by LLNL for commercial processing of UHCS to be carried out by North Star Steel. This processing would produce ultrafine microstructures in sheet, plate, and bar products. Hot-microhardness values were obtained up to 800 C by LLNL for alloys to be processed by North Star Steel. The hardness values provided a measure of the relative resistance to rolling. Due to the emphasis at North Star Steel of concentrating first on solving the continuous-casting problem, these processing tasks were delayed and had not been completed by the completion of the Project at LLNL.

Following the two unsuccessful attempts of casting a commercial 100-ton heat of UHCS on the vertical caster at North Star Steel, the decision was made to make any additional runs using the horizontal casters at First Mississippi Corporation in Holsoppe, PA. Sections of two 1000-pound billets that were produced on the continuous horizontal caster were evaluated and processed to produce a SP microstructure. One composition was based on the standard low-aluminum UHCS, the second was based on a high aluminum-UHCS. The high-aluminum composition showed severe coring and centerline cracking while the low-aluminum was relatively sound. A number of sections were cut off from the two billets, and the cast structure was broken down by hot working. Segregation and stringers of eutectic carbides were still present. North Star Steel indicated that the introduction of magnetic stirring during casting should eliminate this problem. The hot-rolled sections of both compositions were subjected to several different types of thermomechanical-processing procedures. The processed materials were evaluated for various properties: superplasticity, ambient-temperature hardness and strength, solidus temperature, and transformation temperatures. Extensive metallography was performed before and following hot working, on the as-processed conditions, and following SP testing. The SP properties and the coarsening tendencies for the continuous-cast UHCSs were comparable to the results obtained for the ingot-cast UHCSs of similar compositions.

As another alternative to strand castings, North Star Steel cast the molten metal remaining from two failed 40-ton casting trials into 13,400-pound ingots, with three ingots from each heat. One of these large-diameter ingots (20 by 35 by 90 inches) was sent to RMI Company for forging in order to break down the cast structure and to produce 5.25-inch-diameter bars containing a SP microstructure. Forging preforms were to be made from these bars that subsequently were to be used for SP forming demonstration parts at P&W. The first ingot cracked on being reduced to a 13-inch-octagon size, which was attributed to poor procedures. A second ingot was forged using a modified processing schedule. Eight 13.5-inch octagon bars were produced. On subsequent conversion processing, the first bar failed due to unacceptably low processing temperatures; the second bar, with improved temperature control, was successfully reduced to the required final size. Evaluation of sections of this bar by Caterpillar revealed an unacceptable

microstructure due to eutectic carbides and graphite stringers not present in earlier ingots cast with cross sections of 8X8 inch (203X203 mm). The use of this material for the P&W evaluations was stopped. It is likely that additional homogenization and hot rolling steps would improve the condition of the ingots but this was not pursued due to the close proximity of the commercial forming trials by Caterpillar.

Instead, it was determined that Caterpillar would use 500 lb (227 kg) ingot castings as a better simulation of as-cast conditions in strand castings and work on the 40 ton ingot material was terminated. The 500 lb ingot material obtained from Bethlehem Steel was used to produce eight SP blanks (processed under LLNL supervision) to initiate studies by P&W for the Caterpillar demonstration parts. The material was observed to have a microstructure suitable for SP forming and was subsequently made into preform blanks by Caterpillar for the production of the P&W demonstration parts. The carbon content of this material was reduced from 1.6 % to the previous standard of 1.3 %. It was decided that since the lower carbon content was likely to yield the better microstructure, that is, less of a chance in having the unacceptable eutectic carbides and graphitization seen in the large castings, that the next attempt at casting a 40-ton strand casting should be made with a 1.3 % carbon-1.6 % aluminum UHCS.

4. EVALUATION OF SUPERPLASTIC PROPERTIES AND DEMONSTRATION OF FORMING CHARACTERISTICS

4.1 Characterization of SP mechanical properties.

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 $\log s$ 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 above and below 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. The test materials were DETWAD processed by rolling 19-mm (0.75-in.) thick coupons using large reductions per 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). The SEM studies of tested samples revealed the presence of clusters of etch-resistant regions (using a Nital etch) in the "small-strain" materials. These clusters were associated with the presence of low-angle ferrite grain boundaries with an effective grain size of 4 to 6 μm , the ferrite grain size in the well-etched regions being about 2 μm . By contrast, the "large-strain" material revealed well-defined (high-angle) grain boundaries with an as-processed ferrite grain size of approximately 1 μm . The low " m " values are thus largely attributed to the presence of low-angle boundaries which increase the effective ferrite grain size.

A summary of typical UHCS flow stresses as a function of temperature for several strain rates between 10^{-4} and 10^{-1} s^{-1} is shown in Figure 5. The data at 750 °C is typical of a conventionally processed UHCS-low Al composition. Higher temperature data is from UHCS compositions modified to raise the ferrite transformation temperature, A_1 , to above 830 °C (intermediate Al concentrations) and above 900 °C (high Al concentrations). The SP microstructures and properties achieved through modified processing methods and compositions are discussed in section 6.

4.2 Die-filling of subscale parts.

Die-filling tests were performed at Pratt & Whitney (West Palm Beach, FL) and Ladish (Cudahy, WI). Parts were made by forming in the superplastic regime with subscale dies that had been used for evaluating SP bulk forming of commercial components. Parts were forged under isothermal conditions in an argon atmosphere using a 500-ton press at P&W and a 150-ton press at Ladish. A total of 27 parts were made which were scaled-down versions of three different components: a compressor hub at P&W and a high-hub disc and an aircraft-wheel hub at Ladish. The wheel part incorporates a very thin web with some fine details, which should have provided a good test for die filling. Material for two

of the forging preform blanks were processed at Ladish. Material for the remaining blanks were processed at LLNL. All the blanks were machined at LLNL to the required preform dimensions. The presence of an acceptable SP microstructure was assured by examining a cross section from each processed part prior to machining. During Phase 1, two series of parts (11 total) were forged at P&W and one series (7 total) was forged at Ladish. Except for one silicon-containing part (instead of aluminum), these all consisted of low-aluminum UHCSs. The remaining parts were forged at Ladish during Phase II. These consisted of four low-aluminum, two medium-aluminum, and three high-aluminum compositions. Copies of the original data for all these runs were provided to LLNL. A detailed analysis was made at LLNL, whereby the data were plotted in a form allowing comparisons to be made between the different runs. Extensive metallographic analysis was performed on the low-aluminum alloys at LLNL. Most of the metallographic effort on the medium- and high-aluminum compositions was carried out at Ladish.

The first series at P&W were all performed at a true stroke rate of 0.06 (6 %) per minute at 750 C for the aluminum-containing parts and at 850 C for the silicon-containing part. No major differences were observed from these tests either in the forging loads or in the final dimensions of the forged parts. Except for the lack of sharpness in some of the details, the configuration of the dies were well reproduced. Very fine flashing was formed on die closure. In the second series at P&W, four tests were performed at 750 C. Three of these tests were run at different stroke rates of 0.024, 0.06, and 0.6 per minute. Loads were displaced to increasingly higher values with increases in the forming rate. The fourth part was formed at two stroke rates, changing from 0.6 to 0.06 per minute at 85 % of the total stroke travel. Following the rate change, the load approached the values obtained for the part formed entirely at the lower rate. No significant visual differences were observed between any of the parts in these two series of runs. Measurements, however, showed that the height was slightly greater and the diameter was slightly less for the two parts formed with the highest rate. The relatively higher resistance of the material to flow at the higher forming rates resulted in a corresponding increased resistance to die filling.

The first two series of runs made at Ladish covered a temperature range from 732 to 788 C. Some austenite was present at the latter temperature, it being about 13 degrees above the lower (A_1) critical temperature. Three stroke rates were used: 0.05, 0.5, and 5.0 per minute. The effect of forming rate on the loads was similar to that obtained in the P&W tests. Unfortunately, due to the loads reaching the press limit prior to die closure, such closure was not attained in any of the tests. Thus, with the lower loads developed at the lowest rate, significantly more deformation and, correspondingly, more die filling were attainable at the lowest forming rate prior to reaching the press limit. As a consequence, there was a significant difference in the measurements and definition between the finished parts formed at the lowest and two highest rates. In plotting load-versus-travel curves, the curves converged together on approaching the press limit for the two

highest rates and, accordingly, there was no significant differences between these formed parts. An exception to this result was seen in the part formed at 788 C at a stroke rate of 0.5 per minute where the loads remained considerably below those experienced by parts formed below the critical temperature at the same forming rate. As expected, this part was subjected to a larger amount of deformation prior to reaching the limits of the press, ending with a somewhat better defined part. Ladish, in their detailed reports on the runs performed in the second series of runs, as well as in the third and fourth series, indicated the absence of die closure in all these runs.

The subscale wheel parts in the third and fourth series of runs made at Ladish were all formed at a stroke rate of 0.5 per minute. The two parts made with the medium-aluminum UHCS were formed at 738 C, while the three high-aluminum-UHCS parts were formed at 738, 799, and 899 C, respectively. Both alloys were forged below their A_1 temperatures. In all cases, the load reached the press limit preventing punch-to-die closure. In their report, Ladish provided photographs of the UHCS parts to be compared with a photograph of the same part previously made with Ti-6Al-4V. The details in the titanium part were more clearly defined than were the same details in the UHCS parts. This could be largely attributed to the UHCSs not reaching die closure. By contrast, with the titanium alloy having a lower SP flow stress than the flow stresses exhibited by the UHCSs, die closure would have been achieved with the titanium part. The details with the UHCS parts became more clearly defined with an increase in the forming temperature. This is consistent with the corresponding decreases in load and increases in total deformation on reaching the limit of the press as the forming temperature is raised.

Metallographic analysis showed that strain-assisted coarsening had occurred during the SP forging runs. The degree of such coarsening varied somewhat through a cross section of a part, indicative of it being sensitive to the amount of local strain. The relative strain at any point would have to be determined by finite element analysis, and this was not done. For the runs performed at Ladish with the low-aluminum UHCS forged below the A_1 temperature, the change in grain size from that present in the corresponding forging blanks ranged from an increase of about 15 % for a part formed at a stroke rate of 5.0 per minute to over 100 % for a part formed at 0.05 per minute. The forging temperature, which for these parts ranged from 732 to 760 C, seemed to have no measurable effect on the coarsening. The degree of coarsening was influenced primarily by the amount of strain at any point which, in turn, is related to the total deformation in the part. The influence of forging rate on grain coarsening becomes increasingly more significant with an increase in the rate. Material with a medium-aluminum content representative of the blanks used for the forging runs were unavailable. Examination of the microstructure of a cross section of the part formed with this alloy suggested that the grain size in regions of high deformation may have become too large to sustain SP flow. Evaluation of the high-aluminum material did not reveal any obvious microstructural variations across a section of the part

formed at 900 C, nor was there any significant difference between this part and material representative of the preform blank. Consistent with these observations, a specimen of this alloy that had been tested for its SP properties did not reveal any difference in the microstructure between the gage and grip regions. It should be pointed out that finer grain sizes were achievable for the low-aluminum than was obtained for the high-aluminum preforms. Ladish reported ASTM grain sizes of 15 and 16 for the high- and low-aluminum UHCS forged parts, respectively. Based on the results of these tests, Ladish concluded that no economic advantage would be gained by the use of these steels for their applications at that time.

4.3 Die-fill modeling and testing.

The demonstration runs made on subscale parts at P&W and Ladish pointed to the need of establishing a means whereby controlled die-filling tests could be performed in conjunction with finite element analysis (FEA) using the properties of the UHCS that would be used in the commercial demonstrations. In addition, it was desirable to develop the capability to predict the ability to form the critical parts of a potential component by SP forming. Two experimental systems were developed, one was a simple setup used for screening purposes only, the other was a closed-die system providing the means to verify the analytical studies. Argon atmospheres were used for both systems.

Two types of tests were performed in the screening studies. One test involved using a punch containing a small machined rectangular 58-degree V groove which, when pressed into a test sample, formed a corresponding V-shaped protrusion. The extent and sharpness of a protrusion provided a relative measure of the die-filling capability. The second screening test was a simple compression test using 1/2-inch-diameter by 1/2-inch-high specimens compressed into thin disks approximately 1/8 inch thick. The curves showing load versus crosshead travel provided a relative measure of formability. The screening tests were also used to evaluate the relative merits of different lubricants and to select those that could be used with the closed-die system.

The screening tests revealed that a reversal in the general trend of a decrease in applied force with an increase in temperature was obtained if the A_1 temperature was exceeded. This may have been due to an increase in the rate of grain growth caused by the dissolution of carbides and/or to the increased strength of austenite over that of ferrite (in the three-phase region). By contrast, however, such reversal was not obtained for a similar low-aluminum composition of UHCS that contained AlN dispersoids. This alloy was tested at temperatures up to about 125 C above its A_1 temperature. Both the V-groove and disk tests were performed at different loading rates and at various temperatures for different compositions.

A preliminary LLNL design of the closed-die system was reviewed with personnel at Ladish and their suggestions were incorporated into the final design. Testing could be performed up to 950 C. The die-and-punch components were made of TZM molybdenum and were designed to accommodate different configurations. This was facilitated by having both the punch and the die platform each separated into two parts with the configuration components being readily replaceable. Following a number of iterations between FEA and die configurations, a V-shaped ring was selected as the initial configuration of the indenter to be held in the punch. The test blank consisted of a ring (1 inch o.d., 0.5 inch i.d.) having a rectangular cross section and was placed between the indenter and the die. The die contained a matching V groove into which the metal was forced to flow. The analytical sequence was conducted using NIKE2D, an implicit, non-linear, finite-element code with a rate-dependent material model. Experiments were also modeled using the ABAQUS code. This alternative modeling served to check on the NIKE calculations as well as to enable more complex material models to be used that could better represent SP behavior.

A low-aluminum UHCS was used for the analytical-related studies. Based on the screening tests, type E boron nitride was selected as the lubricant. The tests were performed all at the same temperature under constant crosshead-travel rates. Three different starting rates were investigated. These corresponded to initial strain rates of 10^{-4} , 10^{-3} , and 10^{-2} per second, the latter being at a rate just above the maximum SP strain rate attainable for the UHCSs. Some tests involved comparisons between specimens containing different microstructures: fine-grain spheroidal (SP), coarse-grain spheroidal (non-SP), and lamellar-pearlitic (non-SP). The die-filling tests also included friction tests involving various lubricants. To avoid distortion of the die components, the tests were stopped on reaching a limit load of 75,000 pounds. This limit occurred at crosshead-travel distances ranging from 0.368 to 0.375 inch with initial strain rates covering three orders of magnitude. For the lowest strain rate tested, the load increased rapidly from 15,000 pounds to the limit load over a final travel distance of only 0.02 inch; i. e., 80 % of the load increase occurred during the final 5.3 % of travel as "die closure" was approached. The load-travel results obtained here with respect to temperature and forging rate correspond closely with those obtained with the P&W and Ladish runs discussed earlier. A comparison of tests made with a UHCS alloy in the SP and non-SP conditions showed that the loads were about 50 percent greater for the latter condition throughout most of the travel, but that the loads merged on approaching the limit load set for the test machine.

Complete die filling was obtained at the lowest strain rate. The deformed shapes along with contours of the effective stress at various stages of deformation were modeled using numerical calculations. As predicted by the numerical simulations, the extent of die filling decreased with increased loading rates. The experimental load-travel test results agreed reasonably well with the numerical simulations for the intermediate loading rate, despite the need to estimate values for the friction coefficient and some material properties. Nevertheless, the results

of these studies showed the merits of using this system for predicting die-filling capabilities of various configurations.

4.4 Superplastic forming of a commercial component

The part to be superplastically formed under conditions that would demonstrate the techno-economic advantages for the commercial production of UHCSs was established by Caterpillar. The SP forming of the demonstration part was carried out at P&W. The numerical analysis addressing the design of the dies for making the part was initially a joint effort between Caterpillar and LLNL, with P&W performing the SP forging trials necessary to verify the numerical simulations. Compression tests on the alloy to be used for the demonstration part were performed by Concurrent Technology Corporation and funded by Caterpillar. A significant amount of effort had been expended by LLNL on this problem involving both experiments and modeling, as well as including a meeting with members of the analytical group at Caterpillar. For the type of forming operations needed, however, the computational code used by P&W had been proven to be the best available. Also P&W had extensive experience in modeling and metal forming of complex shapes. Thus, in the Caterpillar-P&W agreement, funded by Caterpillar, both the modeling and forming runs were performed by P&W. For this reason, further interaction with LLNL was limited largely to the processing, evaluation, and preparation of the material for the forging runs.

The shapes for the forging blanks were defined by P&W. A number of blanks with the required SP microstructures were to be provided by LLNL. Initially, these blanks were to have been produced from a continuous-cast UHCS. With such material not being available, ingot castings obtained from Bethlehem Steel were substituted. These were processed at Coulter Forge & Steel under LLNL specifications. The ingots were hot rolled to bar, spheroidized and then warm rolled to develop an SP microstructure. The first series of eight blanks were prepared at LLNL and sent to Caterpillar. Subsequent blanks were prepared by Caterpillar, although the plates used for a number of these blanks were processed at LLNL to develop the SP microstructure. All of the blanks were forwarded to P&W for evaluating the forging parameters and for producing the demonstration part. Preforms for the SP forging tests were made from the SP bar using high strain rate forging in the SP temperature range and subsequent machining. The SP forming properties required as input to the DEFORM analysis were obtained from standard laboratory compression tests within the SP temperature range.

Caterpillar executed an Agreement for Services with the forging company to demonstrate the technical feasibility for SP forming a complex diesel engine component from UHCS and to use the results to determine economic feasibility. A cost model determined the path for achieving the economic goal. Both 2-D and 3-D Finite Element Structural Analysis determined that UHCS would meet the strength, life, and thermal specifications for the component.

The Finite Element Method program was used to optimize the preform geometry, the final forged shape and the tooling design. The DEFORM model simulation was very beneficial in predicting the material flow and in highlighting potential troublesome areas. The DEFORM tooling stress analysis capability prevented expensive iterative tooling failure and rework that would have occurred without analytically forging the component. The simulation studies on forging the demonstration part indicated that the critical part of the design should be achievable. These studies, however, indicated that one-stroke forming would not be possible due to the high stresses generated; therefore, multiple die sets had to be used to complete the part.

The diesel engine components were isothermally forged at SP forming rates in a 500 ton vacuum press using the preforms described above. The results demonstrated that the technology to produce complex parts of UHCSs using superplastic forming has been achieved. The results from the demonstrations also indicated that a significant improvement in the superplasticity of UHCS is required for economical forging of these complex diesel engine components. Therefore, further SP forging development work was suspended by Caterpillar for these components.

5. CASTING DEVELOPMENT AND DEMONSTRATIONS

The target composition for the first strand cast at North Star Steel was UHCS-1.6Al-1.35C-0.5Si (with nominal amounts of impurities). This 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, differential thermal analysis (DTA) and dilatometric studies were initiated at LLNL.

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 added 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 as excessive mold friction.

Before continuing with additional castings at North Star Steel, a laboratory-size heat was made at Inland Steel on their experimental continuous straight-mold caster. About 500 pounds of metal from the second NSS heat was remelted and poured into the caster through a tundish to produce successfully a three-inch-square billet about fifteen feet long. Inland removed a section of the billet for their standard analyses. The remainder of the billet was delivered to NSS for chemical, micro, and macro analyses. Center segregation of carbon with excessive amounts of carbides and the customary center shrinkage were observed. These were considered largely avoidable by the conventional application of magnetic stirring commonly used at North Star Steel. Except for this center region the billet was sound and a good fine pearlitic microstructure was present. Two small lengths of the billet were sent to LLNL. One length corresponded to the region where an oil lubricant was used during the startup period and a second length where a powder lubricant was subsequently used. Cross sections of both pieces were microscopically examined confirming the observations reported by NSS. Both pieces showed differences in surface quality with the surface exposed to the powder lubricant being smooth with visible oscillation marks. The surface exposed to the oil lubricant, however, was rough.

Instrumentation of the casting process and a data-acquisition computer-controlled system provided considerable information on the casting parameters. A feed-back loop allowed the casting speed to be controlled in order to maintain the melt zone within predetermined limits. The only significant difference observed between the plain-carbon steels, which are the steels usually cast in the Inland Steel caster, and the UHCS was the relatively high frictional forces recorded between the mold and metal surfaces for the latter material. With input from the Inland casting results, NSS has spent considerable time on analyzing the various casting parameters and focusing on the problems that would have to be corrected in order to strand cast successfully a full heat of UHCS.

Insufficient strength and ductility in the strand cast shell at high temperatures was considered as one potential contributor to the tendency for shell breakout during the solidification process. Strength and ductility data were therefore obtained with a GLEEBLE test machine at LLNL for several different temperatures up to the nil-ductility temperature. Tests were performed on samples taken from a 1090 strand-cast billet and from a UHCS ingot casting for which the composition corresponds closely to that used for the second strand-

casting heat. The tests showed that the UHCS did have relatively good high-temperature strength and ductility, comparable to that of commercially cast 1090 steel. For later determinations of temperature limits, the GLEEBLE nil-ductility test was replaced by a relatively simple nil-strength test. Sample compositions were loaded with a negligible finite weight and heated until failure occurred. The temperature at which this took place was taken to be the solidus temperature. These tests also helped to clarify any ambiguous interpretations that were presented by the DTA results. Data were obtained on compositions that were candidates for continuous casting. In addition, the tests were repeated for material obtained from billets of two of the alloys (the standard low-aluminum and a high-aluminum UHCS) from continuous-cast 1000-pound heats produced at First Mississippi Corporation in Holsopple, PA on a horizontal strand caster. Material from these two castings were also subjected to extensive metallographic and SEM elemental analyses (EDX) in the as-cast condition and following various stages of processing. Hot-hardness values of ingot-cast and continuous-cast materials were obtained and compared. Processed materials were also tested for SP properties. This effort was to determine if any differences existed between the processing of the ingot-cast and billet-cast materials.

Following the two unsuccessful attempts of casting a commercial 100-ton heat of UHCS on the vertical caster at North Star Steel, the decision was made to make any additional runs using the horizontal casters at First Mississippi Corporation in Holsopple, PA. A horizontal caster would be more forgiving than a vertical caster in accommodating the differences in properties between the UHCSs and conventional steels. Prior to making a commercial-size heat on a 40-ton caster, six trial runs were first made with 1000-pound heats, three each with the standard low-aluminum UHCS-1.6Al composition and with a new high-aluminum composition which displayed enhanced potential for SP forming. Successful casting runs were obtained with the low-aluminum alloy, although the chemistry of the first run was off. Material from the last of these runs was sent to LLNL. Less success was met with the high-aluminum material. The first heat broke out at the dummy bar; the second run was completed, but the billet contained internal cracks; the third heat broke out in the mold before the run was completed, producing a casting of only a part of the heat. The cast billets measured 2.4 inches in diameter and billet sections from the final runs of both compositions were sent to LLNL for further analysis and processing.

The results of the trial casting runs suggested that the low-aluminum UHCS should be castable on the 40-ton caster, whereas some modifications would have to be made prior to using this caster for the high-aluminum composition. The decision was made to delay any further effort on the latter material and to proceed with the commercial casting of the former material. Two unsuccessful attempts were made at casting a 40-ton heat of the standard low-aluminum UHCS, which would have provided 5.125-inch-diameter billets. Instead, the molten metal remaining in both of the failed 40-ton heats was cast into 13,400-pound ingots, with three ingots from each heat. The first attempt at strand

casting resulted in mold breakouts in both strands, and this was initially attributed to too high a superheat temperature. Although a corresponding temperature correction was made for the second heat, failures occurred in the form of a premature liquid freeze in one strand and a mold breakout in the second strand. The failure in the second attempt was attributed to insufficient cooling of the molds. Personnel at First Mississippi felt that the failures were due to improper mold taper. They proposed designing new molds to correct this problem. It is likely that the original superheat temperature was not in error. The fact that the smaller billet size was castable, whereas the larger size was not, suggested that there was a size effect that would, in turn, require modification of some of the casting parameters, e. g., heat-extraction rate, mold taper, and lubrication. As a result of the numerous casting failures, it was suggested, and North Star Steel agreed, that a committee of experts was to be identified and that this committee would be requested to establish the cause of these failures and to recommend corrective measures. This remains as an item for North Star Steel to pursue.

6. EVALUATION OF ALTERNATIVE COMPOSITIONS AND PROCESSING METHODS

A preliminary techno-economic analysis by Ladish Co. Inc., which compared the cost of an industrial set of gears produced by traditional manufacturing methods with that produced by superplastic forming, indicated that the SP process may not prove to be economical for the production of these components. The analysis was presented at the Second Technical Exchange Meeting and was based on the SP properties reported at that time for the standard UHCS-low-Al material. As a result of this analysis, and more extensive evaluations of best-case SP properties in the base alloy, the decision was made at the Fourth Technical Exchange Meeting (April 10-11, 1989) that the project at LLNL was to focus primarily on improving the SP properties through both compositional and processing modifications. New goals for SP properties were proposed which would result in at least a break-even cost based on the above analysis. Incorporation of designs which better utilize the potential of near net shape forming estimated that SP forming with UHCS could reduce manufacturing costs by approximately 30 percent.

Following the fourth technical exchange meeting, where the need to achieve the new SP goals was strongly reinforced, a meeting was held at LLNL (June 26-29, 1989) attended by Don Sherman, Mike Strum, Oleg Sherby, and Al Goldberg in order to formulate a matrix of investigations to address this concern. Various alternative thermomechanical processes and compositional modifications were considered which could lead to significant improvements in fineness and stability in the ferrite grain size and in the carbide particle size. These

considerations resulted in the selection of ten additional UHCS compositions for evaluation.

Analyses of existing materials models which describe superplastic flow stresses and maximum strain rates were used to predict the grain sizes and forming temperatures required to meet the new SP goals. Models describing the dependence of minimum stable grain size on the size and distribution of pinning sites (e.g. carbides) were used to define the goals for carbide size and volume fraction. As a result of these analysis, three principal approaches were identified for future developments: 1) develop processing methods to further refine the ferrite grain size, 2) evaluate compositions which raise the peak temperature for SP forming, and 3) evaluate compositions which increase the coarsening resistance of fine-grained ferrite.

6.1 Evaluation of alternative processing methods.

An extensive background on thermal and thermomechanical processing was generated for the standard UHCS-1.6Al composition. To take advantage of this information as a reference point, it was decided to continue using this alloy for evaluating modified or new processing procedures that could lead to improved SP microstructures. New processing methods were evaluated to produce finer carbide distributions, to refine the grain size, and to increase the randomness of ferrite grain orientations with a view towards facilitating superplastic deformation by the grain boundary sliding mechanism.

One processing modification explored was conditioning of the austenite for more efficient conversion of the resultant pearlitic structure into the SP state. The standard UHCS-1.6Al alloy was used for these studies. 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; 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 final grain size of the fine-grained ferrite was not sensitive to the prior-austenite 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. This approach was not pursued further.

In a separate processing modification and as an alternative to the conventional resolutionizing of the pearlitic carbide and reprecipitation as spheroidal carbides, a direct conversion approach was evaluated. Warm-working procedures were examined aimed at decomposing the ultrafine pearlitic lamellae directly into finer spheroidized carbide particles and thereby further reduce the ferrite grain size. In this approach the ultrafine pearlitic carbides break down due to the combined application of temperatures near the A_1 transformation temperature and warm deformation. The degree of decomposition and resulting ferrite grain size was found to depend on the deformation temperature and on the amount of deformation. For a given large amount of deformation, this sub-critical-temperature (SCT) warm work (WW) was more effective in refining the grain size than conventional DETWAD processing. The carbide size distribution and the ferrite grain size were both substantially finer than that achieved by previous methods. Consistent with these observations, strain-rate-change studies showed that both the "m" value and maximum SP strain rate increased in going from a combined DETWAD-SCT-WW process to using solely a SCT-WW process (with equal total WW reductions). The equation frequently used for prediction of microstructural effects on deformation by grain-boundary sliding predicts that the strain rate varies inversely as the third power of grain size. The difference in grain size achieved by the two different processing steps, however, did not influence the change in the maximum SP strain rate as strongly as was expected.

Examination of tested SRC samples revealed that both carbide particles and ferrite grains grew significantly during testing, as shown in Figure 6. Such growth would lead to the observed reduction in the maximum SP strain rates from that expected for the initial grain size. We subsequently heated coupons taken from the same materials used for the SRC tests in order to evaluate the growth that would occur at the test temperature in the absence of any strain. We also examined various areas along a sample tested to failure at the SRC test temperature. These studies showed that, although exposure to elevated temperatures alone did result in some growth, the concurrent strain had a major influence on such growth. As a result of these observations we defined a matrix of heat treatments for a number of UHCS compositions in order to evaluate the compositional dependence of the carbide coarsening rates, described later.

Quench and temper processing of UHCS alloys provided an additional method for the achievement of ultrafine spheroidized carbides. We performed quenching studies on 5 alloys including those containing alloy carbide formers. Quenching temperature was determined to largely control the carbide distributions and morphologies although proeutectoid carbide size was also important. Quenching from low in the austenite plus carbide range produced bimodal carbide distributions, consisting of ultrafine pearlitic carbides and larger proeutectoid carbides. Quenching from high in the austenite range produced fine but continuous carbide networks at prior austenite grain boundaries, with the austenite grains coarsening substantially at the high solutionizing temperatures. Although the as-quenched grain size appeared to be very fine in

some alloys quenched from low in the austenite range, SRC tests resulted in relatively low "m" values. Additional refinement and randomness of the ferrite grains was obtained through a subsequent SCT-WW step. Several SRC tests were made on samples with different tempering treatments which were followed by both the precipitation and SCT-WW steps. The maximum SP strain rates and "m" values obtained for these samples were close to those observed for the large-strain SCT-WW samples.

While pearlitic transformations can be prevented by relatively low cooling rates, such as oil quenching in many in UHCS alloys, it was recognized that this processing method would limit the maximum thickness of SP blanks. One method briefly explored was that of using alloy compositions with increased hardenability including air-hardening compositions. Alloying additions of Ni were especially effective in preventing pearlite formation upon air cooling. The application of these types of alloys was found to be limited, however, by the presence of retained austenite. The retained austenite preferentially converted to extremely fine but pearlitic-type carbides upon reheating to the desired temperature for precipitation of spheroidal carbides. Coarse precipitation at austenite grain boundaries also limited the uniformity of the microstructures produced by this approach. In order to obtain a uniformly fine carbide dispersion it was important to start with a fine austenite grain size with spheroidized carbide particles and to quench from the two-phase austenite-plus-carbide region. The quenching route avoided the formation of any proeutectoid carbide networks and resulted in developing ultrafine submicron carbides.

Quench processing was successful in achieving ultrafine carbide distributions, capable of stabilizing submicron ferrite grain sizes. Ultimately, however, the effective ferrite grain size was determined by its size during SP deformation. Therefore further improvement in the SP properties was limited by the rates of carbide coarsening similar to materials processed by other methods.

6.2 Increases in superplastic forming temperature.

A considerable amount of our effort was directed to examining further alloying of the basic UHCS composition in order to raise the SP forming temperature and thereby achieve increases in SP strain rates and decreases in SP flow stresses. In order to raise the temperature of SP forming and maintain carbide fractions sufficient to stabilize a fine grain size, corresponding increases in the A_1 temperature were required. Both Al and Si additions to UHCS alloys were known to improve ferrite stability, raising the A_1 temperature as shown in Figure 1. Therefore both Si and high-Al additions were evaluated, separately and as co-additions.

Studies at Stanford University [3,4] showed that relatively large additions of aluminum (in the range of 7 to 10 wt.%) to the basic UHCS composition may also introduce new high-temperature deformation mechanisms which differ from

those observed for the UHCS-low-Al alloys. For example, with the UHCS-low-Al alloys, only two distinct regions--one controlled by grain-boundary sliding and the second controlled by slip--are observed on the log stress versus log strain-rate curves based on strain-rate-change (SRC) data. The values for the strain-rate-sensitivity exponent (m) that are indicative of SP behavior fall within the range of 0.4 to 0.5. Two additional regions are observed for the UHCS-high-Al alloys: at strain rates below about 10^{-4} per second " m " values close to 1 can be obtained; at strain rates approaching that set for the SP goals for the project an " m " value equal to about 0.33 is obtained, which is still relatively high. The latter region--related to a solute-drag-controlled slip process-- may be referred to as exhibiting quasi-superplasticity since relatively high tensile elongations can be achieved here. Studies at Stanford University suggested that the new goals for strain rate should be achievable with these alloys forged in the quasi-SP region. In addition, these alloys were shown to be resistant to high-temperature oxidation. The major part of a group of four UHCS-high-Al alloys originally ordered for Stanford University was transferred to the LLNL project. These alloys were obtained from 500-pound ingots that were broken down by hammer forging providing both HW and hot-and-warm-worked (HWW) bar sections. This group of alloys are referred to as Series 3.

A brief study was made on the influence of silicon on the superplastic behavior of UHCS-Al. 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. To compensate for the increases in graphitization potential with Si additions, the Cr levels were also increased. Although it was possible to develop a superplastic microstructure in these alloys by DETWAD rolling, surface cracking was commonly observed during such warm rolling and interpreted as a susceptibility to warm shortness. Furthermore, graphitization occurred in the material containing the higher silicon addition.

Aluminum additions UHCS castings demonstrated the capability of raising the SP forming temperature above 900 °C. The advantages gained in reduced flow stresses through higher SP forming temperatures are shown in Figure 5. The compositions, processing, and testing results on the modified alloy compositions are described later in section 6.4.

6.3 Increases in resistance to microstructural coarsening.

Modification of prior processing steps and the introduction of new processing procedures both led to improvements in the carbide dispersions with ferrite grain sizes of 1 to 2 μm being obtained. Since experimentally based deformation equations showed that the maximum strain rate in the SP regime should vary inversely as either the square or cube of the grain size, it was expected that an increase of about an order of magnitude in the maximum SP strain rate should be

achievable with the finer grain size. Only about a three-fold increase in this strain rate, however, was obtained; this limitation in strain rate arises from the strain-assisted coarsening occurring during the SP tests. To address this issue, coarsening kinetics at 750 °C (a typical test temperature for evaluating SP behavior of the low-aluminum alloys) were measured for eight UHCS alloys (including two high-aluminum alloys and a 3% Al UHCS). The alloys were all quenched from low in the austenite range at a temperature exceeding the A_1 by a fixed increment in order to help normalize the preconditioning of the alloys. Attempts were made to relate the results (ferrite grain size, carbide size, and carbide-volume fraction) to the specific alloying elements and their properties such as their solubility and diffusivity in the ferrite. These efforts clearly identified the size and distribution of carbide particles as the factors controlling the minimum stable ferrite grain size. In agreement with existing models for grain boundary pinning potential, it was determined that finer grain sizes could be stabilized by finer particle sizes and by increased volume fractions of particles. Except for the effect of carbon content, however, which clearly controlled carbide volume fraction, systematic correlations between the microstructural coarsening and particular alloying elements were difficult to isolate. An exception was the general effect of reduced coarsening rates in the more highly alloyed compositions. An example, shown in Figure 7, is the finer carbide sizes in UHCS-3Al-3Cr relative to UHCS-1.5Al-1.5Cr. It was also concluded that improvements in particle stability, sufficient to maintain targeted particle sizes of 0.1 to 0.2 μm during SP processing, will require compositional modifications outside of the range evaluated. On the basis of this information, it was decided to formulate compositions and synthesis methods to introduce new and potentially more stable forms of pinning particles, such as alloy carbides and nitrides, described later.

The limiting ferrite grain size also decreases with an increase in the volume fraction of carbides. Accordingly, the carbon content of the UHCS-1.6Al base alloy was increased from 1.3 to 1.6 wt. %. Although working with a still higher carbon content (but below 2 wt. % C) should further increase the resistance to grain coarsening, the introduction of difficult-to-eliminate coarse eutectic carbides could become a problem. Under laboratory-controlled conditions, however, studies at Stanford and LLNL (through internal LLNL funding) on a 1.8C-1.6Al UHCS did not reveal any evidence of coarse carbide eutectics being present following their processing procedures. With their high carbon content, a ferrite grain size of 0.7 μm was achieved. With this grain size, the maximum SP strain rate was increased by about a factor of three for the 1.8C-1.6Al UHCS over that achieved with the corresponding 1.6C-1.6Al alloy. Strain-assisted grain growth, however, still occurred during SP testing. Maximum carbon contents were limited in practice by the casting conditions and the ability to eliminate undesirable coarse carbides.

6.4 Processing and evaluation of alternate compositions.

Processing and evaluation in Phase II of compositions produced during Phase I. In the latter part of Phase I, ten modified compositions of UHCS had been selected for evaluation. The alloys had been cast as 250-pound ingots by Homer Research Laboratory and consisted of six compositions based on the low-aluminum (1.6-Al) UHCS and four based on the high-aluminum (7.5 and 10 Al) UHCSs. Processing and evaluation studies of these alloys, which were started in Phase I, were continued in Phase II. The selection of the first series of compositions was aimed at increasing carbide stability, the volume fraction of carbides, and/or raising the temperature for ferrite stability. Increasing both the amount and the stability of the carbides should permit a finer as-processed grain size and help retard grain growth during SP forming, thereby increasing the SP strain rate. Elevating the SP ferrite-temperature regime would also result in an increase in the SP strain rate as well as a decrease in the flow stress. The selection of the high-aluminum UHCSs was based on studies at Stanford University that showed that these alloys have a high resistance to oxidation at elevated temperatures and could be formed at higher temperatures with significant improvements in the SP properties relative to the low-aluminum series. Efforts at LLNL were directed mainly on the low-aluminum series, while information on the high-aluminum series was gained primarily from the studies continuing at Stanford University.

In seeking possible solutions to improve the fracture toughness of these steels, two of the alloys investigated contained additions of nickel and molybdenum. Following the breakdown of the ingots, the resulting microstructures of these two alloys were observed to be virtually all austenitic. The high hardenability of these alloys and their low M_s temperatures resulted in the retained austenite. After heating this material for 24 hours to a temperature where pearlite may be expected to form, extremely fine submicron-size carbides were observed in the microstructure. Studies on these alloys were discontinued, however, since they contained excessive amounts of eutectic-carbide networks. In addition, the steels were difficult to process and cut due to the high work-hardening rate of the austenite and the formation of strain-induced high-carbon martensite. Similarly, Sherby found that by quenching the 1.8C-1.6Al UHCS from a temperature completely in the austenitic region, the M_s temperature was depressed sufficiently to prevent the formation of martensite. Subsequent processing of the retained austenite, which included warm working, resulted in the finest microstructure of carbides and ferrite grains that have been obtained. In contrast to the difficulties encountered with the alloys containing nickel and molybdenum, the 1.8C-1.6Al UHCS could readily be processed to develop SP properties.

Several selected high-aluminum (7.5 and 10 % Al) UHCSs were processed and tested at 900 C for their SP properties. The maximum SP strain rates obtained for these tests were almost an order of magnitude higher than the highest rates obtained at 750 and 800C with the low-aluminum alloys. A low-aluminum

UHCS was also tested at 900 C and yielded a maximum SP strain rate similar to the rates obtained at the lower temperatures for these type of alloys. Several of the 7.5 % Al alloys were tested to failure at 900 C at a constant true-strain rate, the rate being that proposed as the goal for the Project following the Ladish-Caterpillar analysis made in Phase I. Although these tests were performed above the SP regime, elongations of nearly 200 % were obtained. The corresponding stress levels, however, were about three times the stress proposed as the goal in the L-C analysis. Comparisons between the low- and high-aluminum UHCS alloys were made as to their resistance to exposure to air for four hours at both 900 and 1150 C. The 10 % Al samples exhibited greatly superior resistance to both oxidation and decarburization relative to both the 7.5 and 1.6 % Al samples, with the latter being the least resistant. Results were given in terms of changes in weight of a sample and changes in thickness of any oxide layer that may have formed. In addition, thicknesses of a surface layer consisting of only ferrite were measured, where such layers were present. Such layers would provide a relative measure of carbon loss.

Processing and evaluation of alloys produced during Phase II. In Phase II, a number of new UHCS materials were prepared for compositional screening experiments by arc melting and casting 50 and 150 gm buttons and, in more promising compositions, 20 lb (9.1 kg) vacuum-induction-melted ingots. This extended the range of alloying investigated with the 250- and 500-pound ingots. Following various thermomechanical treatments the buttons were evaluated for hardenability, the presence and retention of eutectic carbides, and the ability to develop an ultrafine SP microstructure. Following this screening process, a number of buttons were cast of each of the more promising compositions. The alloys included the eight compositions selected by LLNL and Caterpillar at the start of Phase IIA.

The studies in the early part of Phase II clearly indicated that the stability of the grain size during SP forming was dependent on the stability of the dispersions that pinned the grain boundaries. Therefore, an effort was initiated towards examining the introduction of dispersions in UHCS that have greater stability than the $(Fe, M)_3C$ carbides, the carbides that are primarily present in the low-alloyed materials. One approach was to examine the effect of having more of the highly alloyed carbides present. Chromium was identified as an especially favorable addition to UHCSs due to its relatively low cost (lower cost per pound than aluminum), retaining a high solubility of carbon in austenite, increasing hardenability, and increasing wear resistance. Thus, a series of eleven chromium-bearing UHCSs ranging up to 12% Cr with carbon varying between 0.5 and 1.5 wt.% were produced by arc melting. These included some of the eight compositions selected together by Caterpillar and LLNL. Evaluation of processed material showed that the addition of chromium facilitated carbide refinement. At 7% Cr, the preferred carbide phase is shifted from the $(Fe, Cr)_3C$ type to the more highly alloyed Cr_7C_3 type.

Other strong carbide formers were also investigated. Although some of these carbides may have shown a greater resistance to growth than the chromium carbides, difficulties were encountered in refining these carbides when they were present as coarse carbides in the as-cast structure. For example, the high-niobium UHCS ingots contained 10- μm size NbC particles. This coarseness persisted throughout the subsequent hot-working and thermomechanical processing stages. A sample of this alloy was heated by rapid electron-beam scans and controlled to melt only a surface layer which provided rapid cooling. The NbC particles remained coarse due to an absence of dissolution in the molten matrix, although the molten state existed for only a few seconds. Because of this difficulty, studies on Nb additions were discontinued.

Two 20 lb (9.1 kg) ingots, one containing 7% Cr and a second containing 2% molybdenum, were cast for more extensive evaluations. In the processing of the Mo-bearing alloy, difficulties were encountered in eliminating coarse eutectics. Because of this problem, as well as considering the high cost of molybdenum, studies on this alloy were discontinued. The studies with both the molybdenum and niobium additions were made on ingot-cast material. It is likely that with continuous casting, using magnetic stirring with sufficiently high cooling rates, the problem of forming coarse carbides would be greatly reduced. It would also appear that acceptable microstructures could be made from powders containing these highly stable carbides. The development of UHCS alloys containing highly stable carbides in order to prevent deformation-assisted grain growth is worthy of further investigation.

Consistent with the introduction of the more stable Cr_7C_3 carbide in contrast to the $(\text{Fe}, \text{Cr})_3\text{C}$ carbide, evaluations following deformation in the SP regime showed that the resistance to deformation-assisted grain coarsening was increased for the 7% Cr UHCS relative to that observed in the low-chromium UHCS alloys. The carbide growth was extremely limited. The carbides were typically 0.2 to 0.5 μm in diameter, whereas the ferrite grains grew to about 1.5 μm . The strain-rate sensitivity exponent, m , which is a measure of superplasticity, was found to be relatively low (0.27 compared to a minimum accepted value of 0.4 for SP behavior). Furthermore, the addition of this amount of chromium resulted in a further reduction of fracture toughness. The alloy was found to be air hardening containing martensite with some retained austenite on air cooling the hot-forged billet. The billet had then been subjected to a warm-working schedule that developed an ultrafine microstructure. Static annealing tests in the SP temperature range had shown that the microstructure was relatively stable and, accordingly, the alloy should have exhibited SP behavior. Subsequent TEM studies revealed, however, that the as-processed ferrite grain boundaries consisted mainly of low-angle boundaries with high dislocation densities, a structure which would not be amenable to SP behavior. Interestingly, the low-angle boundaries converted to high-angle boundaries during the SP testing. The potential for increased resistance to wear, however, should be a consideration for further examination of these alloys. The possibility of

developing SP behavior through modified processing methods which eliminate the high concentration of low-angle boundaries was beyond the scope of Phase II timelines but also warrants further investigation.

Manganese was viewed as potentially beneficial for enhancing both the SP and ambient-temperature mechanical properties. Manganese additions were expected to result in the following benefits: 1) an increase in toughness based on the presence of retained austenite, 2) a greater ease for microstructural refinement due to an increase in hardenability, and 3) the gettering of sulfur. To determine the influence of manganese, three arc-melted ingots were produced. The aluminum content was selected to offset the depression of the eutectoid temperature due to an increase in the manganese; the chromium and carbon contents were balanced to avoid the formation of eutectic carbides. Based on the microstructural evaluations of both the as-cast and as-processed conditions, the type of carbides formed, and the eutectoid temperatures measured, a composition was selected for a 20-pound ingot to be made at LBL. The ingot was thermomechanically processed to either produce various amounts of retained austenite or to develop a SP microstructure. Further reference to this alloy is presented in the discussion of mechanical properties in a later section.

6.5 Methods to obtain ultrafine dispersions.

In attempting to achieve submicron-size dispersed particles, three new approaches were considered. The first approach was to start with small billets of hot isostatically pressed (HIP'd) powders produced from existing UHCS materials. The powders were to be obtained by either atomizing, attriter milling (AM), gas-jet impingement (GJI), or rotating-electrode processor (REP). Most of these powders were to be produced by various vendors. Some powders were to be made at LLNL by AM and REP. The GJI powders were obtained from EG&G. The rapid nature by which these powders were quenched in producing them resulted in submicron carbide sizes. In addition, the low temperatures (<800 C) used to consolidate these powder helped to retain these ultrafine microstructures. Submicron grain sizes were subsequently obtained by thermomechanical processing.

A significant amount of effort was expended in evaluating the powders, both before and after the hot pressing. Several different compositions, including alloys with low-, intermediate-, and high-aluminum contents were to be evaluated. A low-aluminum UHCS produced from both REP and AM powders cracked during processing. These materials contained relatively high oxygen contents. Microstructural analysis indicated that the materials had not been fully densified and that the cracking had occurred along surfaces of the original powder particles. A number of regions that were devoid of porosity contained submicron-

size microstructures and these appeared to be promising for the development of a SP microstructure.

Three different UHCSs, of low-, intermediate-, and high-aluminum compositions were made into powders by GJI. The first alloy contained 1.8% C in contrast to the 1.6% C content of the standard alloy. The resulting HIP'd-and-swaged materials were successfully processed to produce ultrafine microstructures. The processed materials were tested for SP properties. The SP properties were similar to those obtained with the corresponding alloys processed from ingot castings, and again the results could be attributed to strain-assisted coarsening.

A second approach with powders was to produce alloys from elemental powders mixed to give the desired composition. For this purpose, it was desired to obtain different carbides of about 0.1- μm size. Minimum sizes of about 1 μm could only be obtained and accordingly this approach was discarded. A third approach was to introduce ultrafine dispersions other than carbides into the UHCSs, especially aluminum nitride (AlN), and most of the effort with powders involved this approach. Emphasis here was on the standard UHCS composition. The billets formed with either approach were to be subsequently thermomechanically processed in order to develop a uniform ultrafine ferrite grain size. Some work would also involve additions of Al_2O_3 to 52100 alloy with further additions of aluminum and chromium. This proved to be unsuccessful due to excessive agglomeration of the Al_2O_3 .

Nitrogen-atomized powders of the standard low-aluminum UHCS were first obtained from MIT using a spray-casting system. The powders, which should contain dispersions of AlN, were canned and HIP'd and then further densified by hot swaging. The swaged billet was then processed to develop an ultrafine microstructure, which was followed by studies on grain growth. Using the same spray-casting system, spray-cast plates were obtained of the same alloy. The plates were similarly processed and evaluated for grain growth. Some of the first materials received failed during processing. This failure was caused by oxide films which originated from oxygen contamination in the spray-casting unit. The films were not broken up during the processing. Tests for SP properties were performed on some of the subsequently received materials that had been successfully processed. In addition, compression tests were run at SP-forming temperatures using the screening facility discussed elsewhere.

Although ultrafine microstructures of less than one- μm grain size were developed in the materials containing AlN, the SP properties were similar to those obtained with either the ingot- or continuous- cast materials processed to produce similar grain sizes. Deformation-assisted grain growth also had occurred in both the nitrogen-atomized and spray-cast materials. This growth occurred at 750 C, in spite of the fact that the coarsening studies made for this material at LLNL showed that the AlN did neither dissolve nor grow at temperatures of up to at least 1140 C. In fact, in order to be able to clearly identify the nitrides in the SEM,

a sample had to be heated to 1200 C for 24 hours. It became evident that the nitride volume fractions would need to be substantially increased submicron grain-size stabilization and, accordingly, that the corresponding nitrogen content was too low. The MIT material contained a maximum nitrogen content of 0.030 wt.%. An attempt was then made to increase the nitrogen content by nitriding canned MIT powder using an NH_3 atmosphere. Again, nitride fractions were too low to improve the SP properties.

Attempts were then made to further increase the nitrogen content by introducing it directly into the melt. The first attempt was made with a low-aluminum alloy that was remelted in a small crucible and purged by bubbling nitrogen through the melt for ten minutes. The casting was then processed to produce a SP microstructure, but it showed no improvement in the microstructure over that obtained for the alloy without the nitrogen addition. Grain-growth studies were performed at 750 C and AlN-stability studies were performed at 1100 C. The stability of the nitrides was verified, but the resistance to grain growth was not improved. It was believed that a still higher AlN content was required. Two high-aluminum compositions, one with a eutectoid carbon content, the other with a medium hypoeutectoid carbon content, were then prepared by induction melting in a small crucible modified for bottom pouring. The alloys were purged also for ten minutes by bubbling nitrogen through the melt. By removing a plug held in the bottom of the crucible, the metal was quench cast on to a copper hearth. Preliminary analysis showed a significant increase in the nitrogen contents. A nitrogen content of 0.3 wt. % was obtained for one of the runs. This compares with nitrogen contents typically between 0.015 and 0.03 % reported for the MIT alloys. Annealing runs up to 1200 C verified the stability of these nitrides. Much higher nitrogen contents should be obtainable with longer purging periods, using overpressures, and with improved techniques. A patent disclosure on introducing nitrogen into the melt for forming dispersions of ultrafine AlN particles has been submitted by Richard Landingham, Michael Strum, and Paul Curtis [10]. The principles of this method offer a considerable potential for producing UHCS's containing a dispersion of ultrafine stable particles in sufficient quantities to prevent ferrite grain growth; these studies should be pursued further.

7. ENGINEERING PROPERTY MEASUREMENTS AND JOINING 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. 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.

Various properties were evaluated by Caterpillar for a number of low-aluminum UHCSs and a high-aluminum UHCS that were produced and processed under Caterpillar's direction. The properties included resistance to wear, abrasion, scoring, and galling, tensile, fatigue, hardness, fracture toughness, machinability, hot formability, and warm formability. Comparisons were made between the UHCSs and other materials. The Caterpillar alloys were similar to some of the LLNL alloys developed during Phase I. Extensive metallography was performed on the high-aluminum alloy with respect to the Kappa-carbide phase. This phase was present to different degrees depending on the prior heat treatment. The properties that were evaluated for this alloy appeared to be greatly influenced by the presence of the Kappa carbide.

Early in Phase II, LLNL-processed material from one of the modified low-aluminum alloys was evaluated by Caterpillar. Metallography and properties were evaluated for the as-received condition and after a subsequent quench-and-temper treatment. Charpy V-notched, Chevron-notched, smooth-axial-fatigue, smooth-tensile, and hardness values were obtained. As additional compositions and processing procedures were developed by LLNL throughout Phase II, the corresponding materials were sent to Caterpillar for their evaluation. In these interactions, emphasis was placed on the standard 1.6Al-1.6 C UHCS.

Evaluation of properties and/or microstructures of new compositions and/or following various processing steps was continuously being performed by Caterpillar. This was done for both the LLNL materials and the materials being processed under the guidance of Caterpillar. The latter involved material produced both by Bethlehem and by First Mississippi Corporation through North Star Steel. Material from the North Star Steel 1000-pound heats were processed into SP plates by LLNL and subsequently were made into blanks by Caterpillar for the P&W studies. The Bethlehem-related effort includes evaluation of material hot-rolled at Bethlehem that was produced for the Caterpillar P&W demonstration parts.

Microhardness tests were carried out at LLNL between 25 and 800 C on several alloys, amongst which were alloys that were to be processed by North Star Steel. The hardness-temperature curve for the North Star Steel low-aluminum material was found to be close to that obtained for the ingot-cast material of similar composition. Surprisingly, the corresponding curves for the high-aluminum materials differed from each other in that at temperatures above 100 C, the ingot-cast material had lower values than those obtained for the continuous-cast alloy. Furthermore, the values for the high-aluminum continuous-cast material were similar to those obtained for the low-aluminum materials. These discrepancies

may have been due to experimental problems. Additional material was requested from North Star Steel in order to repeat the measurements. Hardness values measured on a high-aluminum UHCS made by P/M, which was obtained from Kobe Steel, were similar to those observed on the corresponding ingot-cast material. Hardness measurements were also made on a 1090 steel obtained from North Star Steel. The values for this steel were considerably below the values obtained for the UHCSs. The alloys were initially either in the pearlitic or spheroidal condition.

Evaluation of fracture toughness. Three approaches were used to investigate means of improving fracture toughness (FT): 1) developing retained austenite, 2) increasing fineness of the microstructure, and 3) reducing the alloying content. The retained austenite was obtained by raising the manganese content. A 20-pound ingot was produced at the Lawrence Berkeley Laboratory for this purpose. The alloy was processed to produce a SP microstructure, as well as various amounts of retained austenite. The amount of retained austenite was controlled by the severity of the quench (oil versus air) and the austenite quench temperature. The alloy was essentially all austenitic when quenched from the highest temperature. The maximum toughness was observed when intermediate amounts of austenite were detected.

One of the UHCS alloys produced during Phase I, having intermediate amounts of aluminum and chromium, was selected to evaluate the benefits of ultrafine microstructures on FT. The processed material of this alloy included plates that had been ausformed to produce a microstructure consisting of pancake-shaped prior-austenite grains decorated with carbide networks and a uniform distribution of ultrafine eutectoid carbides. Having this anisotropic microstructure and assuming that the pancake shapes would act as crack arrestors, the alloy was also used to determine differences due to notch orientation. The FT tests showed that specimens notched in the crack-arrestor orientation required about a 20 % increase in load over the crack-divider orientation. The increase in toughness was associated with delamination, and the prior austenite-grain boundaries were considered as the source of this delamination. This suggests that UHCS laminates may be one of the methods for increasing FT of these steels. Two additional alloys having a reduced alloying content in order to decrease the volume fraction of carbides were also evaluated. In addition to these alloys, tests were performed on materials having the standard low-aluminum UHCS composition.

Because of the limited thickness of the processed materials, the single-edge-notched bend test (SENB) was used for the FT tests. In order to obtain a valid K_{IC} value, a fatigue crack would normally have to be introduced in the specimen. Unfortunately, several samples failed during the fatigue cycling. It was therefore decided that machined starter notches would be used. A chevron starter notch was selected to assist in arresting the crack upon being initiated at the machined

notch. This provided a precrack for FT determinations. A crack was then propagated beyond the chevron notch for the FT determination. The test results gave only relative values referred to as K_Q . In addition, a number of the early tests were performed with specimens having machined V notches instead of the chevron notch. Caterpillar used a plane strain chevron notch for their FT studies (ASTM E 1304). They reported K_{Iv} values that were significantly lower than the K_Q values calculated at LLNL. This discrepancy is due to differences in specimen size and loading rate. LLNL specimens were thinner than those used by Caterpillar and were loaded at a reduced rate of stress-intensity (K) increase.

In addition to the tests being performed at LLNL, a number of sections of LLNL-processed materials were sent to Caterpillar for their evaluation of FT. The materials consisted of the three alloys referred to above. Several other materials, including the low-alloyed material, were processed as sheet or plate and were also sent to Caterpillar for evaluation of various other properties.

Joining studies. The feasibility of pressure bonding UHCSs has previously been demonstrated by Sherby at Stanford [5-7] and by Lesuer and Syn at LLNL [8,9]. They formed laminates of UHCSs by both press bonding and roll bonding in the temperature range for SP forming. For the Project, two commercially available joining processes, diffusion bonding (DB) and friction welding (FW), were evaluated for joining SP UHCS components. Diffusion across the bonding interface is greatly enhanced by the high density of grain boundaries present in the ultrafine microstructure. Because of the relatively low temperatures (below A_1) that can be used for DB, the process should have only a very limited effect on the parent-metal microstructure. Furthermore, the DB structure would undergo minimum distortion and have low residual stresses. The major disadvantage of DB is that the surface has to be free of oxides in order for diffusion to occur. Thus, it is common practice to use an interlayer to protect the bonding surfaces from oxidation. The softer interlayer also helps to flatten the surface asperities, increasing the metal-to-metal contact area.

Surface chemistry and roughness, which are important in DB, are of minor importance with FW. The frictional heat and applied force upset the mating surfaces sufficiently to provide a clean interface for welding. However, because of the relatively high localized heating and high localized forces, a heat-and-deformation zone is created at a friction-welded bond.

Joining of UHCSs was successfully achieved with both processes. Low-, medium-, and high-aluminum UHCSs were included in the test matrix. The welds for the low-aluminum UHCSs demonstrated strengths comparable to those of the base metal. Relatively low elongations were first obtained with the FW specimens. The elongation was subsequently increased to near the parent-metal values by annealing below the A_1 temperature; yield strengths, however, were reduced. The diffusion bonding was evaluated both without and with the

use of interlayers. The deposit was made by either electroplating (about 10 μm thick) or physical vapor deposition (25 μm thick). These preliminary tests, which showed promising results, were designed as an exploratory study to determine the feasibility of commercially joining UHCSs.

8. BASIS FOR SELECTION OF COMPOSITIONS FOR COMMERCIAL DEMONSTRATIONS

The effect of aluminum additions on the processing and properties of UHCS was extensively investigated by both LLNL and CAT. Based on these investigations, an optimum aluminum content was selected as 1.6 Al. The UHCS-1.3C-1.6 alloy is referred to as the standard UHCS-low-Al material. The criteria for selecting this aluminum addition were based primarily on the ability to develop ultrafine microstructures and corresponding SP properties. The presence of aluminum greatly reduces the extent of proeutectoid carbide networks and results in a corresponding simplification of the primary processing steps (formation of fine pearlite), and a minimum aluminum content was required to obtain acceptable microstructures. The aluminum additions, by decreasing the eutectoid transformation rates upon cooling, also facilitate achieving through-thickness uniformity in the secondary processing steps (formation of ultrafine ferrite grains and carbide particles). SP forming temperatures can also be increased due to increases in the A_1 transformation temperature with aluminum additions. The above considerations favor an increase in aluminum content. By contrast, it was expected that increasing aluminum contents would lead to greater difficulties in strand casting these materials. In addition, the potential for decarburization during thermomechanical processing (TMP) increases with an increase in aluminum content in the low-aluminum range. Results of investigations and analyses of these criteria were presented and led to the selection of a specific aluminum content for the low-aluminum materials.

In the first two progress reports we described studies for establishing the critical phase boundaries and transformation kinetics pertinent for specifying the processing parameters. Evaluations were made of the microstructural observations and SP-test results following various DET (divorced eutectoid transformation) and DETWAD (DET with associated deformation) steps. The degree of spheroidization was observed to be strongly influenced by the temperature range over which the transformation took place. We noted that "large-strain" deformation resulted in an increase in both the maximum SP strain rate and strain-rate-sensitivity exponent, m , over that obtained from "small-strain" deformation used in the DETWAD process. A number of studies were performed to determine the influence of finishing temperature on the austenite grain size obtained during hot working (HW), on the resulting pearlitic microstructure, and on the subsequent as-processed SP microstructures. A wide range of austenite grain sizes were observed. However, neither the amount of

deformation of the austenite nor its grain size showed any measurable influence on either the pearlite morphology, pearlite fineness, or the final ferrite grain size. While the additional HW did result in improved uniformity, hot working in certain temperature ranges could introduce some coarse proeutectoid carbides. Studies were also made on the standard UHCS-low-Al alloy with two different silicon additions. The main advantage of silicon is that it is more effective than aluminum in raising the A₁ temperature. The presence of silicon additions, however, resulted in graphitization and in a tendency towards warm shortness during rolling.

Theoretical predictions based on existing empirical relations suggest that the SP strain-rate (10^{-1} s^{-1}) and stress (5 ksi) goals proposed by Ladish should be achievable with a ferrite-grain size of about $0.5 \mu\text{m}$ providing that no grain growth takes place during the SP forming operation. Processing methods have now been developed to achieve average grain sizes close to this value. For example, in evaluating some low-aluminum UHCSs, which were obtained from Bethlehem-produced ingots that contained 1.7 and 1.8 % carbon, average ferrite grain sizes of about $0.8 \mu\text{m}$ were developed. A corresponding maximum SP strain rate of 8×10^{-3} per second was obtained, a rate which is significantly below that predicted by the theory. This discrepancy is due to grain growth having occurred during the test. For a 1.8 % carbon low-aluminum UHCS, with an average ferrite-grain size of $0.74 \mu\text{m}$ and an average carbide size of about $0.25 \mu\text{m}$, a SP strain rate of 2×10^{-2} per second was reported by Don Lesuer and Chol Syn (at LLNL, independent of the Project). Significant grain growth was also observed after testing of this alloy. The problem thus centers on preventing such grain growth from occurring. It appears that the SP strain rates achieved in Phase II may be the upper limits for UHCSs in which the grain-size stability is based on carbide pinning of grain boundaries, at least for the $(\text{Fe},\text{M})_3\text{C}$ type carbides, and when the alloys are synthesized using either ingot or continuous casting. Until the problem of deformation-assisted grain growth could be solved, it was decided that the basic low-aluminum (1.6Al-1.6C) would be used to demonstrate the ability to commercially continuous cast, process, SP form a commercial component.

As another alternative to strand castings, North Star Steel cast the molten metal remaining in two failed 40-ton heats into 13,400-pound ingots, with three ingots from each heat. One of these large-diameter ingots (20 by 35 by 90 inches) was sent to RMI Company for forging in order to break down the cast structure and to produce 5.25-inch-diameter bars containing a SP microstructure. Forging preforms were to be made from these bars that subsequently were to be used for SP forming demonstration parts at P&W. Evaluation of sections of this bar by Caterpillar revealed an unacceptable microstructure due to eutectic carbides and graphite stringers not present in earlier ingots cast with cross sections of 8X8 inch (203X203 mm). The use of this material for the P&W evaluations was stopped. It is likely that additional homogenization and hot rolling steps would improve the

condition of the ingots but this was not pursued due to the close proximity of the commercial forming trials by Caterpillar.

Instead, it was determined that Caterpillar would use 500 lb (227 kg) ingot castings as a better simulation of as-cast conditions in strand castings and work on the 40 ton ingot material was terminated. The 500 lb (227 kg) ingots were observed to have a microstructure suitable for SP forming and were subsequently made into preform blanks by Caterpillar for the production of the P&W demonstration parts. The carbon content of this material was reduced from 1.6 % to the previous standard of 1.3 %. It was decided that since the lower carbon content was likely to yield the better microstructure, that is, less of a chance in having the unacceptable eutectic carbides and graphitization seen in the large castings, that any future attempts at casting a 40-ton strand casting should be made with a 1.3 % carbon-1.6 % aluminum UHCS.

9. ECONOMIC ANALYSIS AND BUSINESS PLANS

A preliminary techno-economic analysis performed in Phase I, which compared the cost of an industrial set of gears produced by traditional manufacturing methods with that produced by superplastic forming, indicated that the SP process may not prove to be economical for the production of these components. The analysis was presented at the Second Technical Exchange Meeting; it was based on the SP properties reported at that time for the standard UHCS-low-Al material. As a result of this analysis, together with the difficulties encountered in attempting to successfully strand cast a heat of UHCS, the decision was made at the Fourth Technical Exchange Meeting (April 10-11, 1989) that the project at LLNL was to focus primarily on improving the SP properties through both compositional and processing modifications. New goals for SP properties were proposed which would result in at least a break-even cost based on the above analysis. In a further techno-economic analysis made on an existing complex component it became clear that many desirable features were absent in the design of this component. Using conventional manufacturing methods, however, the costs would become excessively high if these features were incorporated into the design. By modifying the design of this component, many of these features can readily be introduced through SP forming. This was illustrated at the Fourth Technical Exchange Meeting and was further expounded at a subsequent briefing to the DOE (December 4, 1989). This later economic analysis, which is based on achieving the new SP goals and on the current cost of steels, showed that SP forming with UHCS can reduce manufacturing costs by approximately 30 percent if the component were designed to take advantage of SP forming.

Business plans for SP UHCS's were prepared by two consulting firms, Engineering Systems, Inc. in Aurora, IL and Crow Contract Engineering Services

in Peoria, IL. Engineering Systems was selected by LLNL during Phase IIA, while Crow Contract was selected by Caterpillar during Phase IIB.

Engineering Systems contacted a number of technical organizations and manufacturing companies and considered a number of potential applications in the automotive, aerospace, railroad, construction, and industrial-equipment industries. They pointed out that in making the analysis they were handicapped by the lack of available economic and property data for the production and use of UHCSs. Because of the superior combination of strength and ductility of the UHCSs over conventional steels, applications for sheet and plate products should exist for structural requirements such as in the transportation industries. A proven cost-saving application as a driver sheet for SP forming of titanium at Rohr Industries was identified. In identifying various applications and components where UHCSs might be used, they pointed out the techno-economic considerations that would have to be evaluated before acceptance of these steels. The market for the high-aluminum compositions was not investigated; however, the potential use for these alloys in engine exhaust systems, where high-temperature oxidation resistance would extend service life, was mentioned. They felt that probably the best opportunity for the commercialization of UHCSs would be for components with features that are introduced to improve the characteristics of the component and designed to take advantage of their SP forming properties. The factors that had to be considered in making a techno-economic analysis were discussed. Except for the driver sheet example and for a hypothetical analysis made of substituting UHCSs for gray and ductile cast irons, quantitative information, which would be necessary in arriving at any definitive conclusion as to the techno-economic viability for the commercialization of UHCSs, was absent.

The business plan presented by Crow Contract Engineering Services focused primarily on a market strategy to be taken for the successful commercialization of UHCSs and the costs of the corresponding operations required in carrying out this strategy. A corresponding schedule of events to implement this strategy was presented. A main target would be the automotive industry. They indicated the importance of providing samples of the UHCSs to potential users. This would also result in providing valuable feedback. They indicated five different companies that were interested in evaluating or learning more about the UHCSs. There was a need to identify sources for producing UHCS rounds and sheet, at least, to provide for test materials. They suggested that since many powder metallurgical (P/M) products have properties similar to those of UHCSs that UHCSs be considered as a potential substitute for such P/M parts. The numerous P/M components used in vehicles were listed. They indicated that the automotive industry would likely be the largest consumer of these steels. In addition to replacing P/M components, automotive applications of other products exists, such as sheet. In comparing UHCSs with several other steels used for automotive applications, the advantages of the UHCSs over these steels were discussed. Using various assumptions, sales and profits were projected for five years through 1998. This profit would be offset by marketing expenses projected for the

same time periods, showing a continuous increase in net profit following a deficit in the first year.

Some of the current efforts involving the Project participants were briefly described by Crow Contract Engineering Services. A number of test and performance studies that were carried out by Caterpillar were listed. The results of these studies, however, were not presented. As with the business plan formulated by Engineering Systems, the lack of real data (in contrast to the assumptions made) prohibits making a credible techno-economic assessment for commercialization of UHCSs.

The need for developing a comprehensive property data base and the need for obtaining input from other industrial sources on their experiences with UHCSs are obvious. The trial runs made at P&W for the Caterpillar part by SP forging of bulk preforms should be followed through with evaluation of other forms to be used for sheet and plate products. The automotive industry, especially General Motors and Chrysler, has expressed interest in evaluating the UHCSs. Furthermore, Chrysler had indicated considerable interest in joining the SP Steel Project, while General Motors suggested that they might be interested in joining and would like to learn more about the Project. The follow-through on having additional participants extend the Project was delayed pending decisions by Caterpillar and North Star Steel.

10. TECHNICAL MEETINGS

Eight Technical Exchange Meetings on Development Of Superplastic Steel Processing were held during the Project; four during Phase I and four during Phase IIA and IIB. Representatives from DOE, LLNL, Caterpillar, and North Star Steel were present at all the meetings. Ladish was represented at four of the first five of these meetings. Dr. Oleg D. Sherby, consultant from Stanford University was present at the first six meetings. The meeting dates and meeting locations are listed in the following:

- First Technical Meeting: May 18, 1988 at Ladish Co., Inc. in Cudahy, WI.
- Second Technical Meeting: August 17, 1988 at North Star Steel in Monroe, MI.
- Third Technical Exchange Meeting: April 10-11, 1989 at Caterpillar in Peoria, IL.
- Fourth Technical Exchange Meeting: August 22, 1989 at LLNL in Livermore, CA.
- Fifth Technical Exchange Meeting: August 9, 1990 at Caterpillar in Peoria, IL.
- Sixth Technical Exchange Meeting: February 12, 1991 at LLNL in Livermore, CA.

- Seventh Technical Exchange Meeting: June 15, 1992 at North Star Steel in Monroe, MI.
- Eighth Technical Exchange Meeting: April 1, 1993 at LLNL in Livermore, CA.

In addition to these formal meetings, frequent communications were held between individuals as well as holding several ad hoc meetings with limited attendance from the participating organizations. A testimony on the SP Steel Program was presented by Caterpillar on April 23, 1991 to the Subcommittee on Environment, Committee on Science, Space, and Technology, United States House of Representatives.

11. FINANCIAL SUMMARY

DOE funding:

Phase I.....	\$1,500,000
Phase IIA.....	706,000
Phase IIB.....	<u>1,820,000</u>
Total.....	<u>\$4,026,000</u>

In-kind contributions by industrial participants:

Costs submitted for Phase I:

Caterpillar.....	\$170,457
North Star Steel....	199,889
Ladish.....	<u>99,732</u>
Total.....	<u>\$470,078</u>

Costs verified by DOE for Phase I:

Caterpillar.....	\$160,650
North Star Steel....	189,889
Ladish.....	<u>83,878</u>
Total.....	<u>\$434,417</u>

Costs submitted for Phase IIA:

Caterpillar.....	\$128,152
North Star Steel....	86,716
Ladish.....	<u>29,826</u>
Total.....	<u>\$244,694</u>

Costs verified by DOE for Phase IIA:

Caterpillar.....	\$128,152
North Star Steel....	80,000
Ladish.....	<u>29,826</u>
Total.....	<u>\$237,978</u>

Costs submitted for Phase IIB:

Caterpillar.....	\$631,951
North Star Steel....	36,123
Ladish.....	<u>5,247</u>
Total.....	<u>\$673,321</u>

Costs verified by DOE for Phase IIB: (unavailable at the time of printing)

Total in-kind contributions by industrial participants for Phases I, IIA, and IIB:

Estimated total costs submitted:

Caterpillar.....\$930,560
North Star Steel..... 322,728
Ladish..... 134,805
Total.....\$1,388,093

Total costs verified by DOE:

(unavailable at the time of printing)

Total relative cost sharing estimated for Phases I, IIA, and IIB

DOE.....\$4,026,000
Industrial..... 1,388,093
Total.....\$ 5,336,719
Industrial/DOE..... 34%

The Industrial-to-DOE costs of 34%, based on the total submitted costs, are above the 30 % cost sharing mandated by DOE. Two of the Phase IIB costs still have to be verified by the DOE. It is expected, however, that after verification of the remaining costs, the cost ratio based on the verified costs will also exceed the required 30 % value.

12. PATENT DISCLOSURES

Three patent disclosures on UHCSs were made by LLNL personnel. The first disclosure deals with a number of alternative thermomechanical processing schemes for developing ultrafine microstructures in UHCSs. It is identified as DOE Case No. S-74323 (RL-11546) LLNL Docket No. IL 8788. A patent application--DOE-US series No. 08/034430-LLNL Doc. No. IL-8788--was subsequently submitted for these processes (Strum, Goldberg, Sherby, and Landingham) and is currently in final office action. The second disclosure deals with the introduction of nitrogen to the melt in order to develop a sufficiently high concentration of ultrafine nitrides on subsequent solidification in UHCSs. It is identified as DOE Case No. S-78,432 (RL-12155) LLNL Docket No. IL-9159 (Landingham, Curtis, and Strum). The third disclosure deals with solid-state welding of UHCSs. It is identified as DOE Case No. S-80,016 (RL-12439) LLNL Docket No. IL-9329 (Anne Sunwoo). Patent applications were not prepared for the latter two disclosures.

13. CONCLUDING COMMENTS

The principal objectives of the project, which were to provide the basis for producing, processing, and forming ultrahigh carbon steel (UHCS) materials on a commercial scale were demonstrated for a basic composition containing 1.6% Al and 1.35% C. In Phase I, aluminum additions to UHCSs were established which provide as-cast microstructures amenable to conversion to SP microstructures and which have been demonstrated as capable of being continuously cast on a pilot plant horizontal caster. Aluminum concentrations which greatly reduce the tendency for the formation of brittle carbide networks were identified, a problem which has largely prevented more widespread commercial application of high carbon steels containing proeutectoid carbides. With the Al additions to UHCS alloys, conventional hot rolling treatments were demonstrated as suitable for ingot breakdown. The characterization of aluminum effects on the Fe-C phase diagram and on the austenite transformation kinetics assisted in the development of appropriate processing schedules for these alloys. Processing procedures which produce uniform SP microstructures in thick section bar and plate were developed and demonstrated.

The new goals imposed by the Ladish analysis on strain rate and stress became the main focus at LLNL during much of Phase II. A goal was established for an increase in the SP strain rate of two orders of magnitude over the maximum SP strain rate obtained in Phase I, as well as maintaining the flow stresses exhibited at the lower SP strain rates. Various attempts were made to achieve this goal with modified compositions and new processing procedures that were aimed at increasing the fineness and stability of the microstructure. Theory and extrapolation of the data indicated that the strain-rate goal should be attainable with grain sizes of about 0.5 μm . The as-processed grain sizes attainable during Phase II have been decreased from about 2 μm to 0.8 μm or less; therefore, SP strain rates close to the goal should have been achievable. Because of the deformation-assisted grain growth, however, which still remained a problem, the expected increase in the maximum SP strain rate was not realized, although some improvement in the strain rate was obtained. Assuming that the proposed strain-rate goal was valid, it became apparent that in order to meet this goal, highly stable particles for pinning the grain boundaries would be needed in an alloy. Considerable effort was expended in attempting to achieve this condition; methods included the introduction of strong carbide formers and using a powder metallurgy approach to obtain the desired structures for evaluation purposes. The incorporation of aluminum-nitride particles obtained by bubbling nitrogen into the melt appears to be promising and this should be pursued further. The AlN particles were shown to be extremely stable at the elevated temperatures. The attempts at increasing the resistance to grain growth with strong carbide formers should also be further investigated, although some of these studies were discontinued due to difficulties in refining some of these carbides. The question remains whether the strain-rate goal set by Ladish based upon hot-die forging practice is a realistic requirement for numerous other applications where

isothermal forging is warranted. Future studies should address a more global approach in considering what industries and applications would benefit from the use of UHCSs.

Only partial success was achieved on continuous casting. Nevertheless, there is a strong consensus that the problems can be rectified without the need to modify the UHCS compositions by making appropriate improvements in mold design, mold lubricants, cooling parameters, casting speed, etc., and that these modifications should be made before attempting additional casts in any future work. Many of these modifications would have to be based on corresponding modeling studies and these studies should be performed with input from a committee of casting experts. The parameters for the commercial thermomechanical processing to produce SP microstructures in sheet and plate have been provided by LLNL for rolling and evaluation by North Star Steel. Commercial forging of UHCSs using either rotary, press, or hammer forges has been successfully performed.

Although the demonstration of SP forming that was carried out at Pratt & Whitney proved to be largely a technical success, and illustrated that a commercial complex part could be made of UHCS using superplastic forming, Caterpillar indicated that the techno-economic analysis showed that SP processing was not competitive with conventional processing in producing the demonstration part. There were certainly valuable lessons learned in this demonstration effort, however, that will be beneficial in pointing out limitations and advantages in the commercialization of potential UHCS products.

Numerous requests have been made for information on UHCSs, especially from the automotive industry. The demand was largely for properties and for sample materials that would allow evaluating new design and processing opportunities that would justify the use of these steels. The higher strength-ductility combination that can be obtained with the UHCSs over that available from conventional steels suggested the potential for reduced weight in using these steels for structural components, such as chassis, door beams, reinforcement rails, frames, suspension components, and bumpers. These components would all be made from sheet or relatively thin plate and, as such, would generally be well in the regime of plane stress. Thus, the low fracture toughness expected for thick sections would be of little or no concern here. The potential of reducing joining costs by SP forming of some of these components was also expressed by the automotive industry. Interest was also shown in exploring the use of UHCS laminates for damping vehicle vibrations. The high damping characteristics exhibited by such laminates were demonstrated by Lesuer, Syn, and Sherby at LLNL (internally funded at LLNL). The large potential savings by the use of UHCSs as a driver plate for SP forming of titanium has been demonstrated by ROHR Industries. Personnel at Macdonnell Douglas Research Laboratories have expressed interest in evaluating the UHCSs for plate applications. In a written communication to LLNL, Inland Steel indicated its interest in the possibility of

using these materials for high-strength-steel applications. The increased forgeability and improved surface finish obtained with these steels as a result of the ultrafine structure has been demonstrated for several different parts forged at conventional high rates below the eutectoid temperature. Interest was recently shown by a tire company in evaluating the use of these steels for radial tires. The interests expressed by a number of companies on using UHCSs in numerous other applications were presented in the business plan prepared by Crow Contract Engineering Services. A recent publication [9] also lists a number of applications for these steels in different industries.

14. SUGGESTIONS FOR FUTURE STUDIES

Additional future studies could enhance the potential for the commercialization of UHCSs. To maximize this potential, these studies should include many of the following tasks: 1) investigate new concepts to further improve the SP properties, 2) develop and identify modifications that may be required in the production and processing the steels for different applications, 3) evaluate applications and forming of sheet and plate products, 4) evaluate applications and forming of laminated metal composites containing UHCS, 5) develop knowledge for high-rate warm forming of UHCSs, 6) evaluate the application of UHCSs for high-strength structural components, 7) perform modeling, friction, and lubrication studies in the forming of UHCSs, 8) develop joining procedures for UHCSs, 9) improve fracture toughness by identifying the fracture mechanisms and the microstructural features that control the fracture and by determining how alloying and particulate modifications and processing variables affect these mechanisms, 10) develop an understanding and a solution for the continuous casting of UHCSs, and 11) develop a database of properties pertinent to the use of UHCSs and improve our understanding of the role and synergism between composition, processing, and heat treatment for optimization of properties.

The development of a broad database on the mechanical, forming, thermal, and corrosion properties of interest to potential users would have greatly facilitated the ability to explore the market for these steels. Mechanical properties that were reported by Sherby for the UHCSs developed before the start of this Project demonstrated the potential for these steels. More recent work by Syn, Lesuer, and Sherby on a UHCS containing 1.8 % carbon showed that a wide range of strength and ductility properties were attainable by using various alternative processing procedures. Some exploratory user properties were obtained by the Project on the new steels being investigated. Now, with the great amount of interest expressed by industry in obtaining information on UHCSs, a matrix of user data should be obtained for these steels in various conditions : 1) containing the as-processed SP microstructure, 2) following simulated SP forming, 3) following various heat treatments to emphasize different user properties and 4) following warm and/or cold processing for sheet and wire products. In addition to providing potential users of the UHCSs with this database, sample materials should be made available to interested users for their evaluation.

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16. LIST OF ACRONYMS

A₁ The temperature at which austenite begins to form in steel upon heating
A_{cm} In hypereutectoid steel, the temperature at which the precipitation of cementite starts during cooling
AM attritor milling.
ASTM American Society for Testing Materials
CAT Caterpillar, Incorporated
CC continuously cast
DB diffusion bonding
DET divorced eutectoid transformation (precipitation of spheroidal vs. lamellar carbides)
DETWAD divorced eutectoid transformation with associated deformation (DET with warm deformation for the purpose of grain refinement)
DOE Department of Energy
DTA differential thermal analysis
EDX energy dispersive x-ray analysis
FEA finite element analysis
FT fracture toughness
FW friction welding
GJI gas jet impingement
HIP hot isostatic pressing
HW hot working
HWW hot and warm working
K_{IC} plane strain fracture toughness
K_Q fracture toughness value (may not meet thickness requirements of K_{IC})
K_{IV} plane strain (chevron notch) fracture toughness
LLNL Lawrence Livermore National Laboratory
MIT Massachusetts Institute of Technology
M_s martensite start temperature
P&W United Technologies Pratt & Whitney, Incorporated
RMI Reactive Metals Incorporated
SCT sub-critical temperature
SEM scanning electron microscopy
SENB single-edge notched bend (fracture toughness specimen type)
SRC strain-rate-change (tensile test method)
TMP thermal-mechanical processing
TTT time-temperature-transformation
TZM a high temperature molybdenum alloy containing titanium and zirconium
UC University of California
UCHS ultrahigh carbon steel
WW warm work

As-Hot-Rolled
Pearlitic UHCS

2 μ m

Conversion-Processed
Superplastic UHCS

2 μ m

Figure 1. Thermal/mechanical processing converts the ultrafine pearlite characteristic of hot-rolled UHCS into a superplastic microstructure consisting of fine-grained ferrite and spheroidal carbides.

Figure 2. Influence of aluminum and silicon on the A_1 (lower austenite transformation temperature) for UHCS-1.25C steel. Numbers adjacent to data points refer to wt.% of the element.

3 °C/min

20°C/min

Figure 3. Post austenitization cooling rates of 3 °C/min result in spheroidal carbide formation while higher rates, such as 20 °C/min result in lamellar eutectoid carbides in this UHCS alloy.

Edge

Center

Figure 4(a). Continuous deformation from the soaking temperature in a press forging results in lamellar eutectoid carbides.

Edge

4 μm

(1/4) t

Center

Figure 4(b). Controlled temperature forging produces uniform microstructures throughout the thickness of the billet.

Figure 5. Flow stresses vs temperature for typical superplastic microstructures at several strain rates. Data was determined from SRC tensile experiments.

As-Processed

750C (1382F)/
90 min.

Figure 6. The ultrafine as-processed microstructure produced by subcritical warm working of a UHCS alloy is significantly coarsened by an anneal of 750 °C for 90 min.

UHCS-1.5Al

(quenched from 810C)

750C/
30min.

UHCS-3Al-3Cr

(quenched from 850C)

750C/
24hrs.

Figure 7. The coarseness of the microstructure is decreased in a UHCS containing a higher alloy content relative to a lower-alloy UHCS subjected to the same anneals.