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THE HISTORY OF ULTRAHIGH CARBON STEELS

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Abstract

The history and development of ultrahigh carbon steels (i.e., steels containing between 1 and 2.1% C and now known as UHCS) are described. The early use of steel compositions containing carbon contents above the eutectoid level is found in ancient weapons from around the world. For example, both Damascus and Japanese sword steels are hypereutectoid steels. Their manufacture and processing is of interest in understanding the role of carbon content in the development of modern steels. Although sporadic examples of UHCS compositions are found in steels examined in the early part of this century, it was not until the mid-1970s that the modern study began. This study had its origin in the development of superplastic behavior in steels and the recognition that increasing the carbon content was of importance in developing that property. The compositions that were optimal for superplasticity involved the development of steels that contained higher carbon contents than conventional modern steels. It was discovered, however, that the room temperature properties of these compositions were of interest in their own right. Following this discovery, a period of intense work began on understanding their manufacture, processing, and properties for both superplastic forming and room temperature applications. The development of superplastic cast irons and iron carbides, as well as those of laminated composites containing UHCS, was an important part of this history.

Introduction

The modern study of ultrahigh carbon steels (UHCS) began in the mid-1970s and continues to the present time. The initial work was carried out at Stanford University and, since the late 1980s, also at the Lawrence Livermore National Laboratory. The various facets of their invention and development are described in 95 papers [1-95], (including six United States Patents); the 17 papers in the proceedings of this conference [96-113], and some other papers in other countries. In this paper, we summarize the major steps in this development, and the key microstructural and mechanical characteristics of

the steels. The original UHCS [1, 3] were plain carbon steels containing 1 to 2.1%C (this corresponds to 15-32 vol.% iron carbide) with Mn as the only alloying addition. Subsequently, a number of alloying additions including Cr, Ni, V, Ti, Mo, Si, P, W, and Al was explored [5, 7, 11]. In either plain carbon or alloyed form, it was found that the steels could be thermo-mechanically processed to produce microstructures with fine spheroidized carbides within a fine-grained ferrite matrix. The processed UHCS possess a unique combination of properties, not found in other materials, that make them well suited for structural applications. Specifically, UHCS can have high ambient-temperature strength, hardness, and ductility, and excellent high-temperature formability via superplasticity. Included in this UHCS work was a substantial amount of work carried out on cast irons and iron carbides. A summary is shown in Table I of the various areas of study within the history of UHCS development and the specific references for each group are indicated. The six patents that have been issued on this work are listed and it is noted that others are planned.

Table I. Subject Areas within the UHCS Study

Area	References
Superplasticity	1, 3, 4, 5, 6, 7, 9, 11, 13, 14, 19, 22, 26, 45, 51, 53, 58, 60, 65, 76, 77, 78, 85, 89, 90, 93, 94
Laminated Composites	12, 20, 29, 33, 43, 44, 46, 54, 56, 57, 59, 61, 63, 64, 66, 68, 69, 70, 71, 72, 74, 91
Cast Irons and Iron Carbides	2, 6, 9, 23, 25, 26, 28, 30, 31, 45, 53, 55, 58, 62, 65, 77, 78, 83, 86
Damascus Steels	10, 15, 17, 18, 27, 32, 37, 41, 42, 50, 67, 81, 82
Processing and Micro-structure Property Relationships	33, 34, 40, 75, 79, 80, 83, 84, 86, 87, 88, 92
Reviews	16, 21, 37, 39, 47, 48, 49, 52, 73, 95
Powder Metallurgy	2, 23, 25, 28, 30, 31, 38
Patents	3, 33, 38, 40, 51, 84
Heat Treatment	8, 24
Other	55, 62

Steels in the UHCS composition range, in addition to having been studied scientifically since 1975, also have an interesting ancient history. In 1979, Sherby and Wadsworth discovered that the typical carbon compositions in the modern UHCS they studied were essentially the same as in Damascus steel swords of ancient times, i.e., from about 1.4 to 1.8% C [10, 15, 17, 18]. In fact, the ancient uses of UHCS compositions are found in a range of weapons developed in several cultures. For example, UHCS are also an integral part of the Japanese sword which was first made in about 500 AD and continues to be made to the present time. Other ancient artifacts also exist that have UHCS compositions, or structures that are comprised of laminated combinations of UHCS with other steels. Included are European and Chinese welded Damascus blades, welded Damascus gun barrels, the Indonesian Kris, and Merovingian (Viking) blades [18, 37, 41, 42].

Some of the key historical events in the development of ancient steel products composed of UHCS are summarized in Table II. In general, it has been acknowledged that ancient steel objects having UHCS compositions are often the result of relatively primitive steel making processes and that they can be fashioned into articles of exceptional sharpness, retention of cutting edge, and toughness, if appropriately thermomechanically processed. In general, this processing has occurred in Asian countries, whereas in Europe, little success was achieved in processing such very high carbon contents.

Damascus steel weapons were renowned for their fine cutting edge and high toughness; that is, they were highly resistant to cracking. Perhaps even more important, they were famous for the incomparably beautiful surface markings which gave the weapon an identity as well as a mystic and spiritual feel. The method of their manufacture by blacksmiths of ancient times is believed to be a lost and forgotten art. Legends abound that Damascus steels were first developed at the lost continent of Atlantis, that they were brought to India when Atlantis sank, that they had special healing powers, and that they were used by Alexander the Great in his conquest of the civilized world. These fascinating tales led us to investigate the history of Damascus steel making. In the course of this study, a successful effort was made to reproduce such markings on UHCS materials and the published procedure has been described as the modern rediscovery of Damascus steel making [67, 81, 82].

An example of a Damascus steel sword (a Persian scimitar) is shown in Fig. 1. The special surface pattern is a swirlly distribution of the proeutectoid carbides (the white areas) achieved by a complex forging procedure. These white regions are different from, but related to, the iron carbide network also shown in Fig. 1. In the case of the special sword shown in Fig. 1, the vertical arrays, known as "Mohammed's ladder," arise from locally introduced variations in the different directions of upset forging.

It is believed that the Damascus steel was made in India where it was known as wootz. (The origin of the name of "Damascus" is attributed to European traders.) The steel was widely traded in the form of castings, or cakes, that were about the size of hockey pucks. The best blades are believed to have been forged in Persia from such Indian wootz; this wootz was also used to make shields and armor. In Persia, they were known as "poulad Janherder."

Table II. Key Events in Ancient UHCS

- 323 BC	Alexander the Great receives a gift of Indian "wootz" from King Puru.
- 300 BC to ~1850 AD	India/Middle East/Europe - Development of Damascus Steel for swords and other instruments of war.
- 400-500 AD to Present	China/Japan - Development of the Japanese Sword.
- Ca 1100	Saladin the Saracen warrior demonstrates the sharpness and toughness of Damascus Steel to Richard the Lion-Hearted.
- 1612	Dimitri Pozharsky and Kuzma Minin defeat invading Poles. Their achievement is memorialized in a statue of them in front of St. Basil's Cathedral, Moscow.
- 1795	Pearson presents his work on "wootz" (Indian steel) to the Royal Society.
- 1824	Bréant - In France, describes equilibrium cooling of a hypereutectoid steel for the first time.
- 1820	Faraday works with a cutler, Stodart, on wootz, but incorrectly attributes its unusual characteristics to small amounts of silica and alumina.
- 1830	Pushkin publishes his poem on bulat.
- 1841 - 1843	Anosov - Publishes his career work "O Bulatah". His statue in Zlatoust in the Urals is the only known statue of a metallurgist.
- 1868	Tchernov - Manufactures Damascus Steels and lectures on Anosov's work in Moscow.
- 1896	Howe publishes his overview of the influence of carbon on ductility in steel.
- 1904 - 1906	Belaiew - inspired by a lecture by Tchernov, leaves Moscow, lectures and experiments in London, and publishes his results.

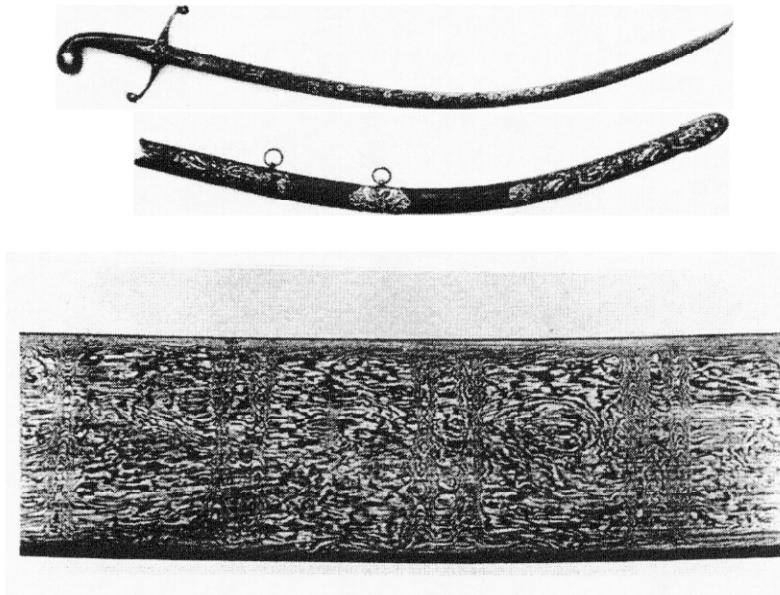


Figure 1. Persian scimitar dating from the 17th century or later in the Metropolitan Museum of Art, New York. The white areas are from aggregations of coarse cementite particles. The local effects of forging are recognizable as vertical arrays in this unusual form of a damask known as Mohammed's ladder.

These steels were also known in the middle ages in Russia where they were called "bulat" steels.

Recently, while in India, one of the authors (O.D. Sherby) discovered a painting of King Puru of India greeting Alexander the Great (about 330 BC). Part of this painting is reproduced in Fig 2. This painting is in the guest



Figure 2. A painting from a mural in Ranchi, India, depicting King Puru of India presenting gifts to Alexander the Great. One gift is an Indian wootz sample contained within the gold box.

house of the largest R&D steel laboratory in the world, the Steel Authority of India, in Ranchi (with a staff of 1200 research scientists and support personnel). After King Puru was defeated by Alexander the Great in battle, (as shown in the painting), the King gave, as a token of respect, his sword to Alexander, and behind the King, his aide is seen to be carrying an additional gift, a gold container within which is a cake of Indian wootz. At the time, this steel was more prized than gold. In a more recent period, the Russian poet, Alexander Pushkin immortalized "bulat" with a similar comparison, when he wrote, in 1830, the following poem:

All is mine, said gold
All is mine, said bulat
All I can buy, said gold
All I will take, said bulat

The exact procedures used by the ancient blacksmiths in making the surface markings on genuine Damascus steel swords (it is termed 'genuine' because it is made from a single ultrahigh carbon composition casting) have been the source of much speculation. When procedures are described, they are usually given in vague terms, with no precise descriptions of temperature of forging, of the cooling rate prior to and after forging, or of the degree of deformation given at each step. In 1979, a specific procedure was proposed [18] which may have been used by the ancient blacksmiths and has become known as the "Wadsworth-Sherby" method [67]. The procedure utilized is a rolling process involving three key steps.

First, the wootz (in this case, an UHCS containing over 1.5% C) is heated to near its incipient melting point (a white heat - 1200°C) to develop coarse iron (austenite) grains. Second, the wootz is cooled very slowly, over a period of several hours, to form a thick continuous network of iron carbide at the boundaries of these coarse iron grains. At this point, surface markings are visible to the naked eye as shown in a modern UHCS in Fig. 3. Third, the wootz is heated to a color between blood red and cherry (i.e., about 650 to 800°C), a temperature at which the iron carbide network remains mostly intact, and the wootz is then mechanically worked extensively to break the network into individual, coarse iron carbide particles that may be spherical or elongated. At this point, the network is no longer continuous, but remains visible as a layered structure, and is very appealing to the naked eye.

Photomicrographs of a UHCS-1.8C material, processed by the "Wadsworth-Sherby" method, are shown in Fig. 4. The low-magnification, optical photomicrograph on the left shows the proeutectoid carbide network with coarse dimensions of about 0.2 mm by 2 mm. It appears to be continuous but, in fact, is not; rather, it consists of broken-up carbides. The background matrix within and adjoining the carbide network is dark upon etching and unresolvable at low magnification. When the carbide network and the matrix are viewed at high magnification, with the scanning electron microscope, the structure is seen to consist of fine iron (ferrite) grains with fine spheroidized carbides; depending on the final temperature of mechanical working, the matrix can be fine pearlite.

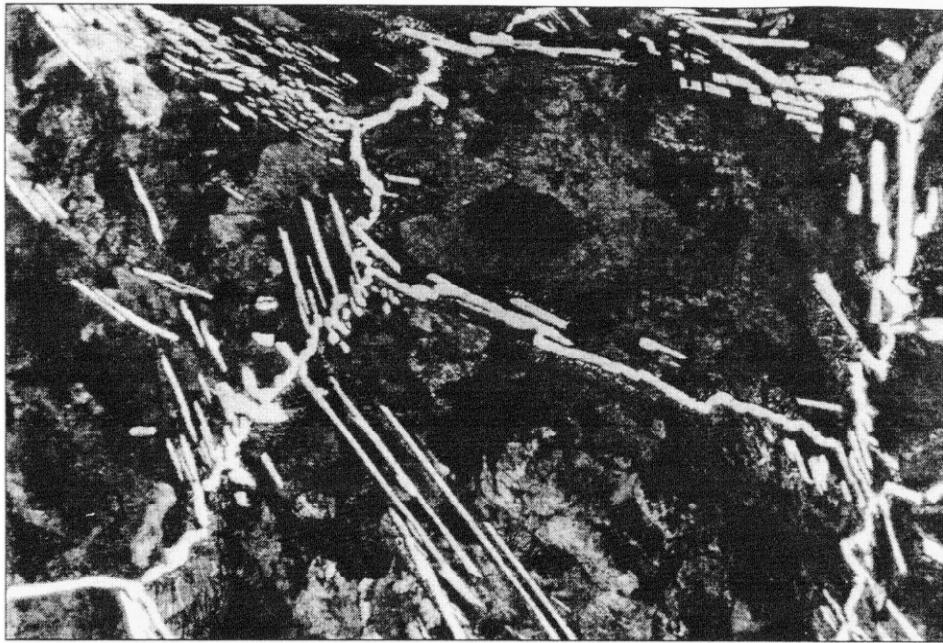


Figure 3. Micrograph of a modern UHCS clearly showing a cementite network. The background structure is pearlite, i.e., alternating layers of essentially pure iron and iron carbide. (Magnification about 200 diameters.)

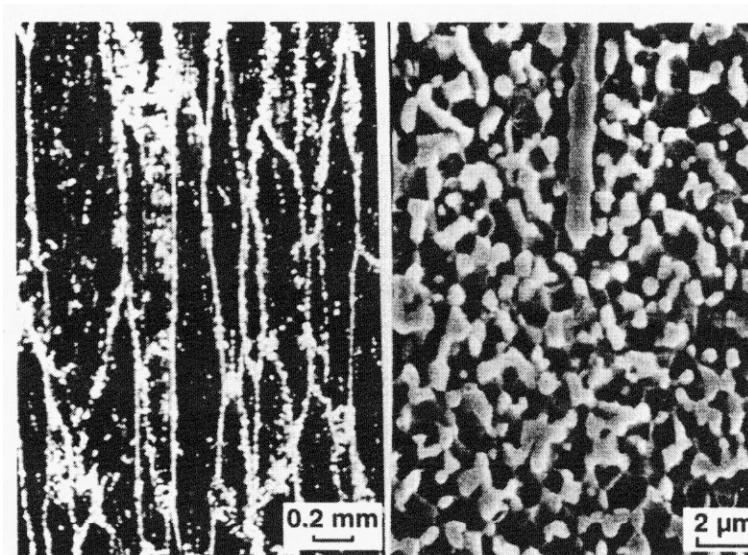
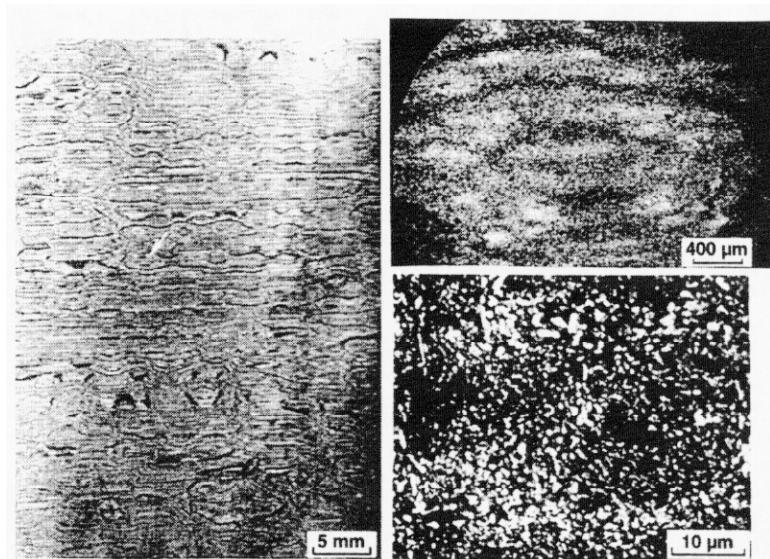


Figure 4. Photomicrographs of a UHCS-1.8C material, processed by the Wadsworth-Sherby recipe. The photomicrograph on the left shows the proeutectoid carbide network with coarse dimensions of about 0.2 mm x 2 mm.

Small changes in the processing procedure for making visible damasks can lead to wood-like patterns in the UHCS-1.8C material as illustrated in Fig. 5. Photomicrographs are shown at three different magnifications. On the left is a low magnification photo showing the wood-like pattern. In the top right, at an increased magnification, the dark etching bands are the broken-up proeutectoid carbides. The highest magnification photomicrograph, lower right, shows the bands of alternating coarse and fine carbides.



UHCS-1.8C with Wood-like DAMASK

Figure 5. Damask on a UHCS-1.8C material processed by rolling to obtain a wood-like structure. Three magnifications are shown illustrating the severe break-up of the proeutectoid carbides in band-like regions.

In order to evaluate whether or not genuine Damascus steels with markings could exhibit superplasticity, tension tests were performed at elevated temperature on the UHCS-1.8C material containing the visible damask that was shown in Fig. 4. The material composition was 1.8% C, 1.6% Al, 1.5% Cr, 0.5% Mn, and balance iron. The flow stress-strain rate response of the UHCS material at 750°C is shown in Fig. 6. The material was found to be

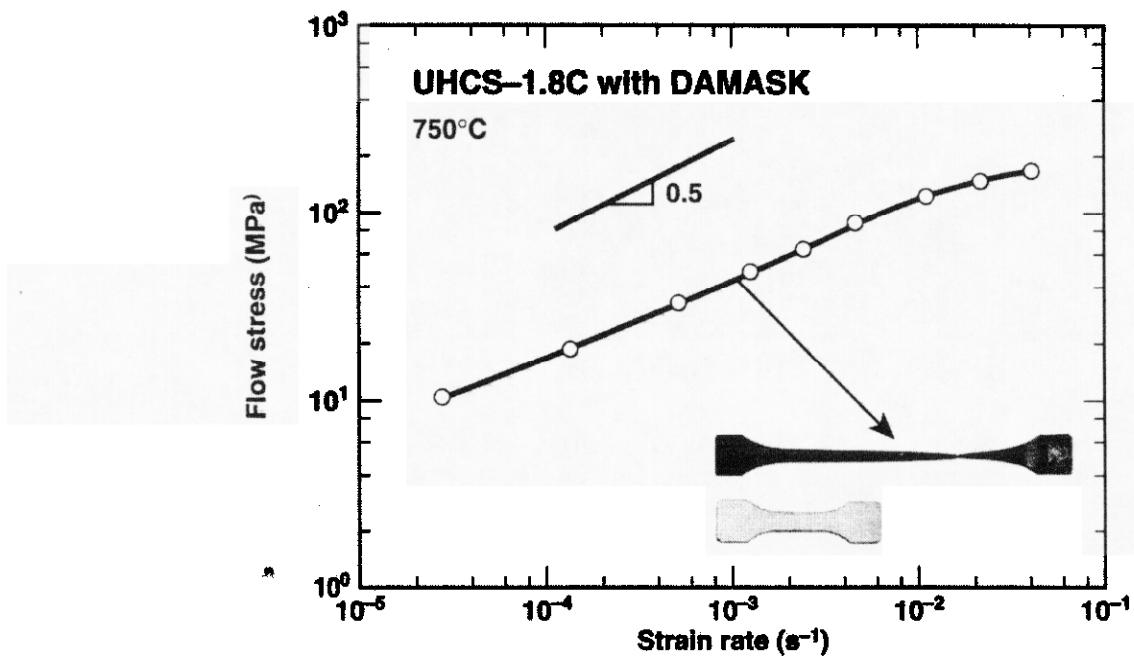


Figure 6. The flow stress-strain rate response of the UHCS material with visible damask at 750°C. The slope of the stress-strain rate curve shows a superplastic strain-rate sensitivity exponent of 0.43. The sample was tested at a strain-rate of 2% per minute to an elongation of 450%.

superplastic, with the slope of the stress-strain rate curve showing a strain-rate sensitivity exponent of 0.43. In the same figure, a sample tested at a strain rate of two percent per minute is shown to exhibit an elongation of 450%. These results suggest that the ancient Damascus steel weapons could have exhibited superplastic characteristics near the A_1 transformation temperature.

The room temperature mechanical properties of the UHCS material depicted in Fig. 4 exhibited a yield strength of 920 MPa, an ultimate strength of 1,145 MPa, and a total elongation of 12%. This is a remarkable combination of strength and ductility and confirms the general statements made about the malleability of ancient Damascus steels.

These steels, now designated as UHCS, have been viewed for most of this century as belonging in the "no man's land of carbon steels" being sandwiched between the extensively-utilized high carbon steels (0.6 to 1.0% C) and the mass-produced cast irons (2.1 to 4.3% C). This is depicted in Fig. 7 which illustrates key stages of the historical development of the most famous phase diagram, the binary Fe-C system. It took many years to complete this diagram beginning with the work of Tchernov of Russia (1868), followed by Sauveur of the United States (1896), by Roberts-Austen of England (1897), by Roozeboom of Holland (1900), and completed by Honda of Japan (1920). Even then, the E point (the maximum solubility of carbon in austenite) was

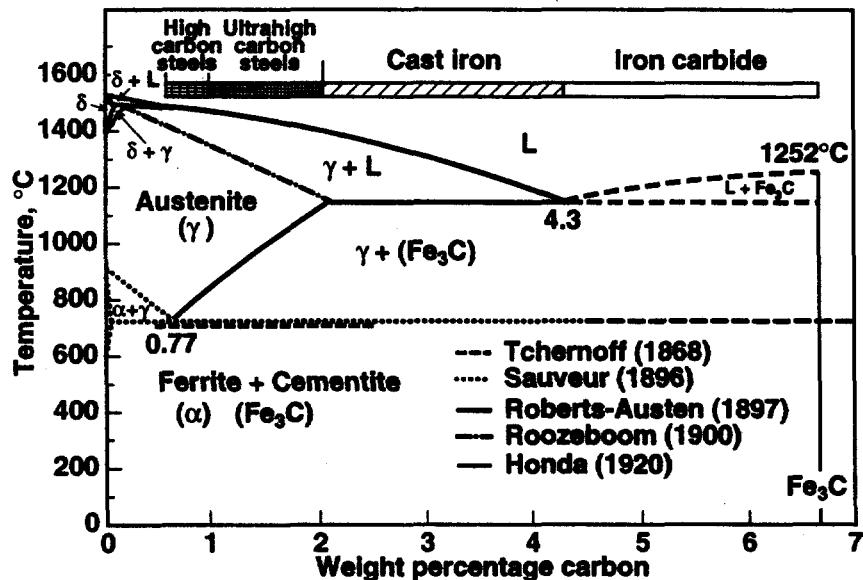


Figure 7. Historical description of the Fe-C phase diagram.

erroneously labeled at 1.7% C and this error was not rectified until 1948 to its correct position at 2.1% C. Beyond 4.3% C (in the diagram of Fig. 7), is the iron carbide region since the majority of the structure consists of iron carbide (65 to 100% from 4.3 to 6.67% C). The specific discoveries on the Fe-C phase diagram are: Tchernov established the A_1 transformation line at 723°C, Sauveur confirmed Tchernov's finding and established the ferrite-austenite region, Roberts-Austen established the austenite-cementite boundary and the variation of the melting point of iron with carbon additions, Roozeboom established the austenite-liquid boundary from thermodynamic reasoning,

and Honda put all the knowledge gained (including the delta ferrite-austenite-liquid range) into a resemblance of the present phase diagram.

Ultrahigh carbon steels are labeled as being in the "no man's land of carbon steels" because UHCS have been considered to be brittle at room temperature and thus have been generally ignored for commercial applications. The origin of this belief can be traced to the classic work of Howe, published in 1891 [114], in which the tensile ductility of steel was studied as a function of carbon content. A key figure from this study is shown in Fig. 8, in which tensile elongation is plotted as a function of carbon content. The tensile ductility decreases dramatically with increasing carbon content and becomes roughly constant, at 2 to 3%, in the region of UHCS. The curve drawn in the figure is that given by Howe. This trend has been accepted historically and has been reproduced in many publications since Howe's compilation. The primary reason for the low ductility in these UHCS is a result of the formation of the continuous, thick network of brittle iron carbide in high carbon steels upon cooling from high temperature to intermediate temperature (for example, from 1000°C to 723°C for a Fe-1.6%C alloy). An example was shown in Fig. 3 of such an iron carbide network for a 1.6%C steel. These thick, continuous networks are locations at which cracks can initiate because iron carbide is brittle at room temperature and cracks within it will readily propagate under stress causing failure in the steel.

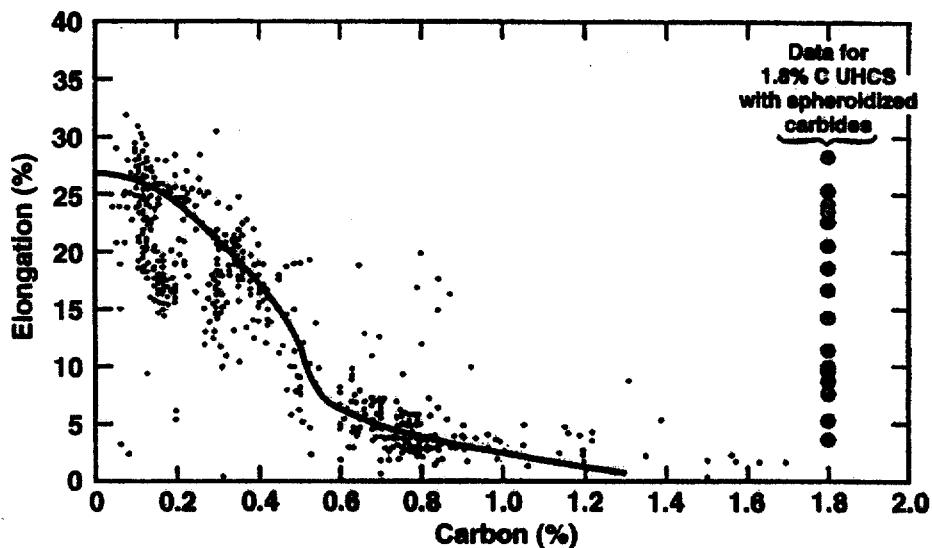


Figure 8. Historical data for the tensile elongation for steel at room temperature dramatically decreases as the carbon increases. (Reproduced from a book by the American metallurgist, Henry Howe, in 1891.) Modern UHCS can have far greater tensile elongations as shown by the data for a UHCS-1.8C.

At Stanford University and at the Lawrence Livermore National Laboratory, procedures have been developed to eliminate the continuous carbide network in UHCS. The result is that relatively-homogeneous structures containing fine, equiaxed, ferrite grains and fine, uniformly-distributed, spheroidized carbides are readily achieved. The range in tensile ductilities for a UHCS-1.8C is incorporated in Fig. 8; the wide range of values shown (from 2

to 25%) reflects different morphologies and strengths produced by various routes.

The modern development of steels based on ultrahigh-carbon contents therefore represents a new and unique approach which is just the opposite of the current trend in steels. Specifically, over the last thirty years, the carbon content in commercially available sheet steels has been decreasing as shown in Fig. 9. These ultralow-carbon steels, that are currently available, have been developed primarily for automotive sheet-stamping applications in which high formability and weldability are of paramount importance. However, reducing the carbon in these steels also reduces strength. As a result, additional strengthening mechanisms, using solid-solution and precipitation hardening approaches, have been required. The UHCS described here contain almost two orders of magnitude more carbon than found in these new steels. They provide a material, however, with high strength, good formability, and promising weldability, a combination of properties that suggest potential sheet applications. Tack welding studies on sheets of UHCS containing 1.5% C show high strength with ductility following a post welding heat treatment.

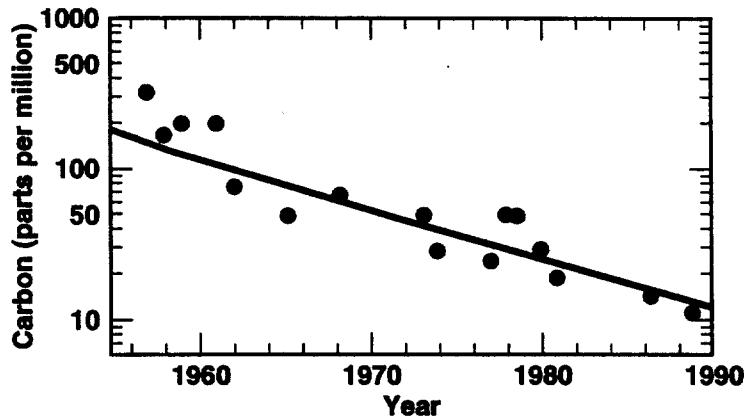


Figure 9. The decrease in the carbon content of automotive sheet steel as a function of time.

The evolution of UHCS can be described in several different ways. In this paper, their evolution is described from the viewpoint of Composition and Processing, Mechanical Properties (including Elevated Temperature, Superplastic Properties, Room Temperature Properties, Influence of Heat Treatment, and UHCS Laminated Composites), and Potential Applications and Future Directions. A summary of key events in the development of UHCS is given in Table III.

Composition and Processing

As may be seen, a number of different compositions of UHCS has been evaluated since 1975 to the present time. The initial studies were on essentially plain carbon UHCS, that is, compositions involving no significant alloying additions, except for manganese [1, 3, 4]. These steels were found to be superplastic following thermomechanical processing. It is worth noting that, following this discovery, in an attempt to study the superplastic

Table III. History of Superplastic UHCS and Laminates Containing UHCS

1975	Plain carbon UHCS shown to be superplastic.
1976	First UHCS Patent issued (#3,951,697).
1976	Cr additions to UHCS-1.6C lead to 1000% elongation and superplasticity in a commercial 1% C 1.5% Cr 52100 bearing steel.
1978	Carbon contents beyond UHCS, containing 2.1 and 2.3%C, shown to be superplastic.
1979	Heat-treated UHCS show $R_c = 66$.
1979	Superplastic-aided bonding of UHCS to other steels and development of UHCS-containing laminated composites.
1979	Superplasticity developed in cast UHCS structures by thermal cycling.
1981	Superplasticity developed in a commercial UHCS tool steel (01).
1982	Enhancement of UHCS powder compaction by superplastic forming.
1982	UHCS principles applied to rapid solidification processing of white cast iron powders.
1983	Impact properties of UHCS-based laminated composites shown to be outstanding.
1984	Superplasticity demonstrated in UHCS-based laminated composites.
1984	Divorced eutectoid transformation, with and without additional deformation, incorporated in UHCS processing and U.S. Patent issued (#4,448,613).
1985	Scientific American article on UHCS and Damascus Steel.
1985	United States Patent issued for UHCS Thermal Cycle Processing of Powders (#4,492,671).
1985	United States Patent issued for UHCS-Si Alloy (#4,533,390).
1987	UHCS principles applied to eutectic Fe-Cr and Ni-Cr white cast irons.
1988	UHCS compositions containing aluminum patented (#4,769,214).
1989	Superplasticity demonstrated in iron carbide.
1991	Newtonian-viscous superplastic flow in UHCS-Al alloy.
1993	Tensile ductility of UHCS-enhanced by thin layer lamination with brass.
1994	Strength and ductility of spheroidized UHCS sheet.
1995	United States Patent issued for Transformation Processing of UHCS (#5,445,685).
1996	Ingot-processed iron carbide.
1996	High strain-rate superplasticity in UHCS.

behavior of plain carbon UHCS, in a more scientific and fundamental manner, high purity Fe-C alloys were prepared and processed. Walser, Kayali, and Sherby [4] made the unexpected discovery that the high purity Fe-C alloys (1.6% C and 1.9% C) could not be made superplastic. The stress exponent value for the pure 1.6% C alloy was high ($n = 8$), that is, the strain-rate sensitivity exponent was low, about 0.15. The accompanying ductility was normal for a non-superplastic ductile metal (50 to 100% elongation). Electron and optical microscopy revealed that the pure, ultrahigh-carbon, iron-carbon alloys could not be made fine-grained. These results emphasize the importance of the normal additives in steel (such as Mn and Si) in retarding and controlling the growth of grains and cementite particles.

Subsequent studies [5, 7, 11, 40, 51] over the decade from 1978 to 1988 centered on dilute alloying additions including chromium, silicon, aluminum, molybdenum, and nickel. Included in the early part of this period of time were studies on two commercial compositions that were at the low carbon end (i.e., 0.9 to 1.1% C) of the UHCS carbon content range. One was 52100 bearing steel (1% C, 1.5% Cr) [7] and the other was 01 Tool Steel (0.9% C, W) [19]. Eventually, extensive research was subsequently focused on the influence of silicon and aluminum in enhancing the superplastic behavior of UHCS [40, 51]. The basis for selecting these two elements was as follows. The maximum temperature where superplasticity in UHCS can be observed is at the A_1 transformation temperature (723°C for plain carbon steel). Above this temperature, austenite forms and a large fraction of the carbides dissolve so the austenite grains can then grow readily and superplasticity will be inhibited. Alloying elements such as aluminum and silicon stabilize the ferrite phase thus increasing the transformation temperature. For example, for a UHCS-1.25C, the A_1 temperature is increased to 850°C with 4% Si and to 950°C with 10% Al. Both UHCS-Si and UHCS-Al alloys were successfully processed to achieve fine-grains, and patents were issued based on their promising microstructure-superplastic property relations. The UHCS-Al alloys with a high concentration of aluminum (7 to 10% Al) showed the most promising high temperature properties.

The UHCS-high Al alloys were also found to be oxidation resistant, with virtually no oxide scale formation even after exposure at 1200°C for long periods of time [62]. Furthermore, since the activity of carbon in iron is reduced to zero when the aluminum content is at 10%, little or no decarburization is observed with the UHCS-high Al alloys. It was not possible to achieve the same fine grain sizes that were obtained with the lower aluminum content UHCS alloys (about 2 μm). Typically the grain sizes in the UHCS-high Al alloys were in the range from 5 to 10 μm . A sub-micron grain size material could be obtained in such compositions, however, by a powder metallurgy-mechanical attrition route and this approach led to high-strain-rate superplasticity [85].

A summary is given in Table IV of key events in the evolution of compositional changes made during the history of UHCS and the primary philosophy behind each change.

Table IV. Evolution of Compositions in UHCS

Year	Alloying Addition	Alloying Purpose
1975	Plain carbon UHCS	Typical steel alloying additions including Mn
1976	Fe-C alloy	Pure Fe-C approach
1976	Ni, V	Originally believed to stabilize the carbides but found to cause graphitization
1976	Cr	Stabilize carbide composition to avoid graphitization
1985	Si	Expand the temperature range for superplasticity
1988	Al	Expand the temperature range for superplasticity
1988	Mn, Mo	Enhance hardenability
1993	High Al	Avoid decarburization and oxidation and achieve ideal superplastic behavior.

Various thermomechanical processes have been developed to arrive at a range of microstructures including fine-grained spheroidized structures, fine pearlite, fine bainite, and fine martensite. The principal thermomechanical processes employed include: hot and warm working (HWW), isothermal warm working (IWW), divorced eutectoid transformation (DET), divorced eutectoid transformation with associated deformation (DETWAD), and combinations of these processes. Other approaches, including thermal cycling of cast structures and powder metallurgy processing (including rapid solidification processing), have also been demonstrated. Details of all of the various processing techniques have been described elsewhere [3, 14, 34, 35, 39].

Mechanical Properties

Elevated Temperature Superplastic Properties

Superplasticity was originally discovered in 1911, but studied sporadically until the early 1960s [115]. Following an intensive period of interest at that

later time, studies focused on model eutectic and monotectoid systems. Early attempts to develop superplasticity in steels were largely unsuccessful and focused on medium carbon steels, and to some extent, thermal cycling, or internal stress superplasticity.

In the early 1970s, the natural place in the iron-carbon system in which to study superplasticity was the hypoeutectoid composition range utilizing the presence of two phases, ferrite and austenite, as means of achieving fine grains. Since the temperature was above the critical temperature, grain growth occurred leading to only limited success in achieving superplasticity. Another natural place to consider was the eutectoid composition of about 0.8%C. The relatively low volume fraction, ~12.5%, of the second phase iron carbide, however, made it difficult to achieve and maintain the necessary fine-grain size because of grain growth. An increase in volume fraction of second phase, by increasing the carbon content was not considered feasible because of the traditional belief that steels of such high carbon content were unworkable and brittle as described earlier. Instead, some attempts to work with the two-phase region of austenite and ferrite were pursued with some limited success.

The move to increase the carbon content to a level above the eutectoid composition in order to achieve superplastic behavior, however, was the driving force that lead to the development of the modern UHCS compositions. It was discovered that, far from being unworkable, the ultrahigh carbon compositions were forgiving in the appropriately selected temperature ranges and, furthermore, that the resulting fine microstructures were ductile at room temperature.

In all cases, high temperature processing to break up the continuous iron carbide network in cast UHCS results in a more forgiving, i.e., more ductile, material. And, contrary to popular belief, iron carbide is not brittle at intermediate and high temperatures. From 1973 to 1976 [1, 3, 4] network-free materials were developed by continuously mechanically working the UHCS (1.3, 1.6, and 1.9%C) as they were cooled from a white hot temperature (1200°C). This mechanical working (by either rolling or forging) broke up the iron carbide networks as they were first forming during cooling, i.e., at a point at which they were still thin and not fully continuous. In this way, the iron carbide that formed upon cooling was unable to grow and create the thick networks normally associated with UHCS. Examples of the fine microstructures developed in a 1.5%C steel, taken by high magnification transmission electron microscopy, are shown in Fig. 10. The light-colored grains are iron and are sub-micron size, and the dark particles are iron carbide. This material is superplastic at high temperature and, of equal importance, is strong and ductile at room temperature. A patent was awarded for the processing procedures in the development of such microstructures in UHCS [3]. An example of a superplastically deformed UHCS sample is shown in Fig. 11, in which an elongation of over 1000% was achieved at 750°C with no evidence of imminent failure. The strain rate was 200% per minute.

The opportunity for net shape forming using superplasticity is now well established for UHCS [73]. Because of the fine grain sizes developed in these

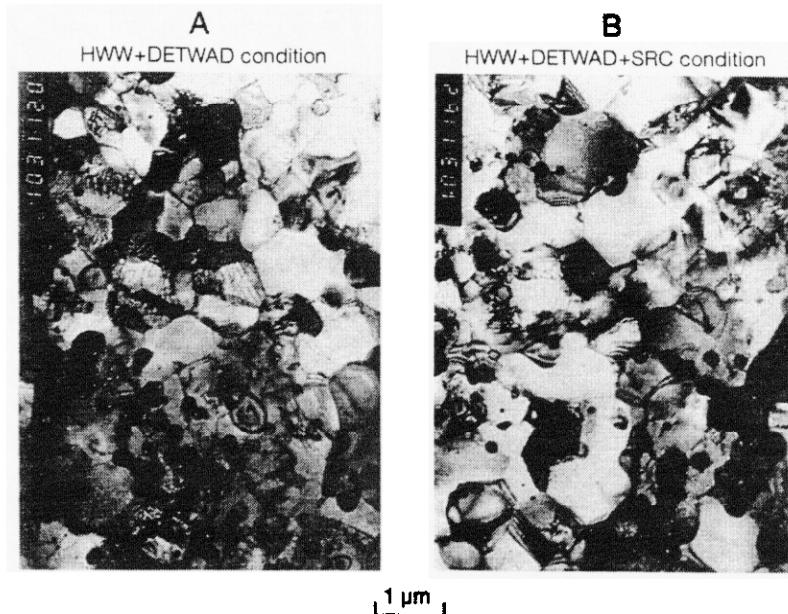


Figure 10. Photomicrographs of a UHCS-1.5C material processed to obtain an ultrafine grain size. The photo micrograph on the left is in the as-processed condition and the photo micrograph on the right is after deformation of the material at 700°C.

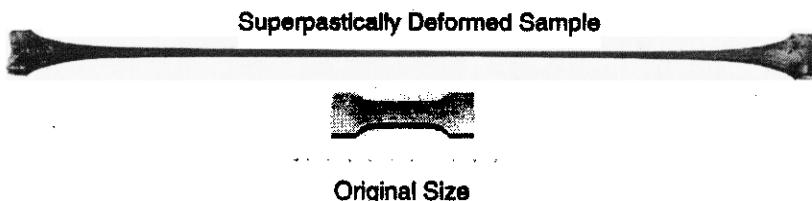


Figure 11. An example of superplastic behavior in a UHCS-1.8C; an elongation of over 1,000% was achieved with no evidence of imminent failure.

materials, elongations in excess of 600% have been obtained in plain-carbon UHCS, and over 1000% in dilute-alloyed UHCS, at warm-working temperatures. For plain-carbon and dilute-alloyed UHCS, the ranges of temperature and composition over which superplasticity has been observed are summarized on the Fe-C phase diagram in Fig. 12. Grain sizes evaluated were typically around 2 μm . For the UHCS-composition range (1.0 to 2.1% C), superplasticity can be observed at temperatures from 650°C-800°C. This temperature range extends above and below the A₁ transformation temperature and thus includes microstructures containing ferrite and eutectoid carbides or austenite and pro-eutectoid carbides. In the austenite-plus-carbide region, the maximum temperature for superplasticity is determined by grain growth kinetics, which results in the loss of the fine-grained microstructure. Grain growth is rapid in the austenite-plus-carbide region because much of the carbide that is present in the initial ferrite-plus-carbide structure is lost because of the high solubility of carbon in austenite. The carbides help maintain a fine-grained microstructure by pinning the

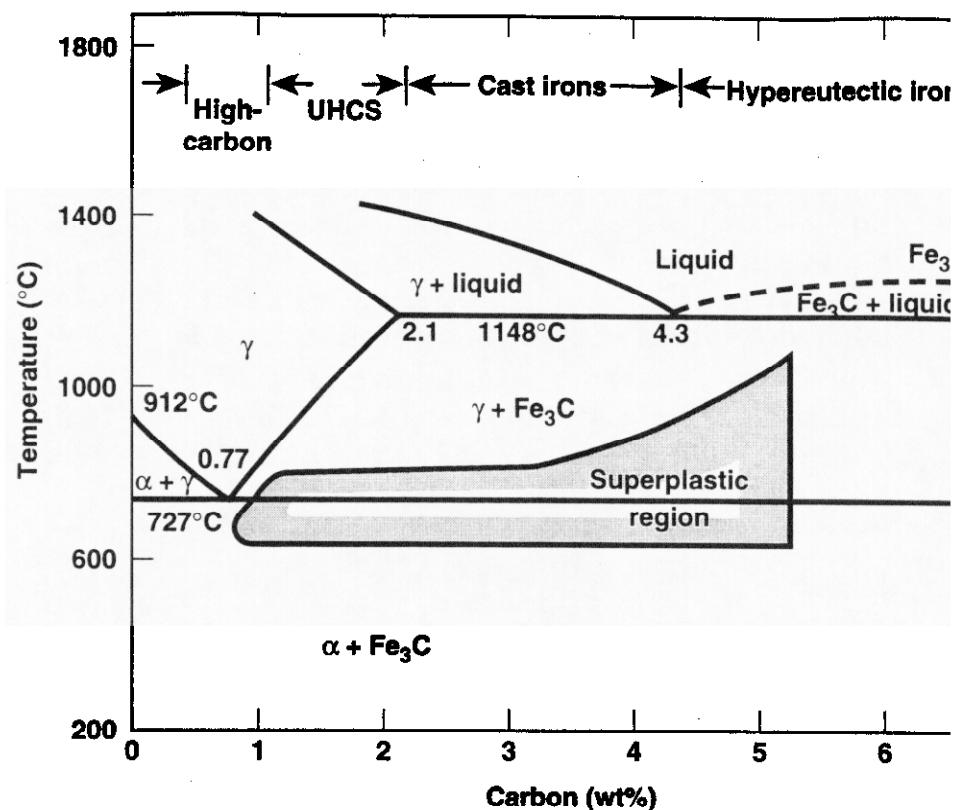


Figure 12. The composition and temperature range where superplasticity has been observed in the Fe-C system.

grain boundaries and retarding the grain-growth kinetics. Superplastic behavior has also been achieved with carbon contents in excess of 2.1%C (Fig. 12); that is, in white cast irons (2.1% to 4.3%C), and hypereutectic irons [2, 6, 9, 25, 26, 45, 53, 65, 77, 78, 83, 86]. For hypereutectic irons, the matrix is iron carbide with discontinuous ferrite and these compositions can therefore be designated as iron carbide materials. The fine equiaxed grains required for superplasticity in these high carbon materials were prepared by powder metallurgy routes, although ingot processing of eutectic and hypereutectic (5.25%C) composition Fe-C alloys also proved to be viable methods for approaching superplastic behavior.

For plain-carbon UHCS, the original maximum strain rates for superplasticity were about 10^{-3} s^{-1} and this condition was achieved just below the A_1 transformation temperature [13]. The maximum strain rate for superplasticity can be increased dramatically through suitable alloying additions such as aluminum or silicon [40, 49, 51, 89], which either (a) permit UHCS to be deformed at higher temperatures within the range of superplastic flow without unacceptable amounts of grain growth, or (b) inhibit the transition from grain-boundary sliding (superplastic behavior) to slip creep (non-superplastic behavior) by raising the flow stress for slip creep. Increased superplastic-deformation temperatures are obtained with these alloying additions because they influence the characteristics and microstructures of UHCS in at least one of four ways; by (a) increasing the A_1 transformation temperature, (b) inhibiting carbide coarsening because the activity of carbon in ferrite is increased, (c) forming complex carbides that resist coarsening, and (d) increasing the amount of proeutectoid carbides.

The maximum strain rate for superplasticity is shown in Fig. 13 as a function of temperature for four different UHCS alloys containing aluminum and silicon. All materials have a common initial grain size of 2 μm . UHCS containing either 3%Si or 1.6%Al both exhibit superplastic flow at a maximum strain rate of 10^{-2} s^{-1} in the temperature range of 770-800°C. Aluminum appears to be much more effective than silicon in raising the superplastic forming rate. The figure also shows the predicted maximum superplastic-forming rate for a UHCS containing 12%Al with a grain size of 2 μm . This material should exhibit a maximum superplastic forming rate of $3 \times 10^{-1} \text{ s}^{-1}$ at 950°C, which is close to commercial forging rates. These results illustrate the excellent potential for these fine grain materials in high throughput, commercial forming operations.

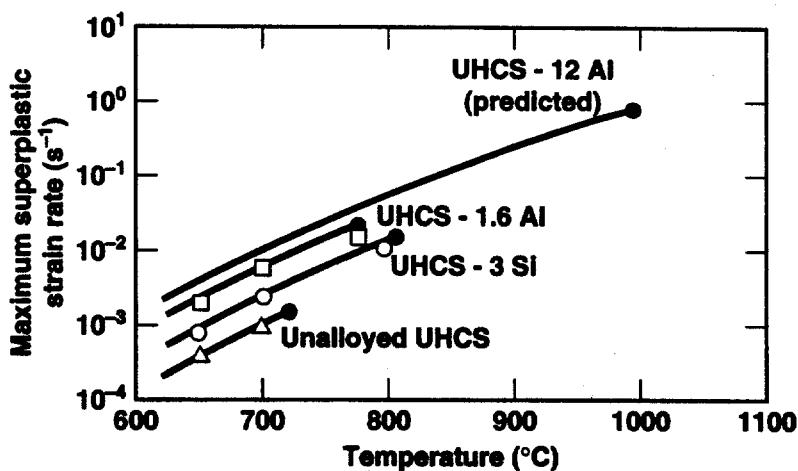


Figure 13. The influence of aluminum and silicon on enhancing the strain rate for superplasticity in fine-grained UHCS. The maximum strain rate for a given composition is indicated with a solid circle.

The strain-rate sensitivity exponent approached unity ($m = 1$) at high temperatures and low strain rates for a UHCS-10Al-1.2C material [60]. That is, the material behaved like an ideal viscous fluid. The results are explained by a modified version of the grain boundary sliding model of Ball and Hutchinson. Fukuyo, et al [60] showed that when grain boundary sliding is accommodated by solute-drag-diffusion-controlled dislocation motion, no dislocation pile-up occurs, and the strain rate becomes a linear function of the stress, i.e., $m = 1$. The measured activation energy for superplastic flow of the UHCS-10Al (200kJ/mole) was in agreement with the model since it equaled the activation energy for diffusion of aluminum in iron.

Since the UHCS-high Al alloys behaved like Class I solid solution alloys (that is, solute drag of dislocations was the rate-controlling process), it was predicted that a strain rate sensitivity exponent equal to $m = 0.33$ would be observed at high strain rates. Such a respectably high strain rate sensitivity leads to quite high elongations, 200 to 300%, and has been termed as "quasi-superplastic" behavior (i.e., resembling superplasticity or being superplastic-like) [115]. This behavior is shown in Fig. 14 in which the flow stress is plotted as a function of the strain rate for three UHCS containing 7 to 10% aluminum. At high strain rates, a strain rate sensitivity exponent of about

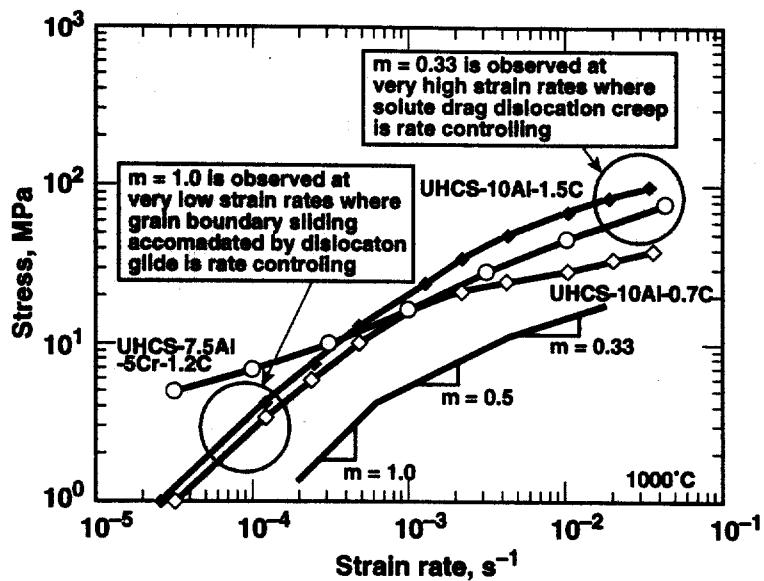


Figure 14. Quasi-superplasticity ($m = 0.33$) and ideal grain-boundary sliding

0.33 was indeed observed corresponding to a rate-controlling process of solute-drag controlled dislocation creep. An elongation to failure of 500% was achieved with the UHCS-7.5Al-5Cr-1.2C material tested at 950°C and at a strain rate of 3% per second. At low strain rates, a strain-rate sensitivity exponent approaching unity was observed. In this case, the mechanism is that of grain boundary sliding accommodated by solute-drag dislocation creep.

An example of a superplastically formed part made from a UHCS-high Al material is shown in Fig. 15. The ring component was fabricated from UHCS-9.3Al-1.23C at 950°C in air. The ring, which is approximately 25 cm in diameter, was superplastically formed in five minutes. Commercialization of this product was intended by Sulzer Brothers of Winterthur, Switzerland, but the project was abandoned because no steel producer was prepared to make the fine-grained UHCS-high Al material. The bevel gear shown in the figure was forged at a conventional forging rate at 650°C, and was made from an unalloyed UHCS-1.25C material. Both components were formed close to net shape and illustrate the excellent die-fill characteristics of fine-grained UHCS [73].

Economical production by bulk forming of UHCS will require three basic conditions. The applied force must be low, typically less than 35 MPa in order to minimize die wear, the strain rate must be relatively high, 10^{-1} s^{-1} or faster, and the temperature of forming should not exceed 800°C, once again to minimize die wear. These were the conditions set by the industrial partners on a government-industry consortium in which Lawrence Livermore National Laboratory was a partner. The three conditions were almost achieved for UHCS-high Al alloys, as shown by the results given in Fig. 16 [95]. This joint program was terminated, however, when it was discovered

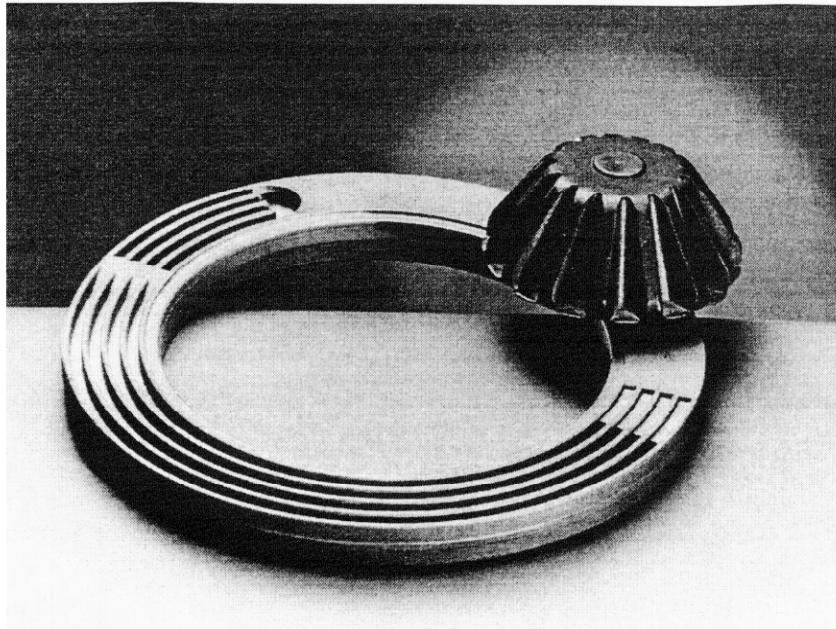


Figure 15. Superplastically formed UHCS components. The ring component (25 cm in diameter) was fabricated from a UHCS-9.3Al-1.25C at 950°C in air in five minutes. The bevel gear was forged at a conventional forging rate at 650°C (the material is a UHCS-1.25C).

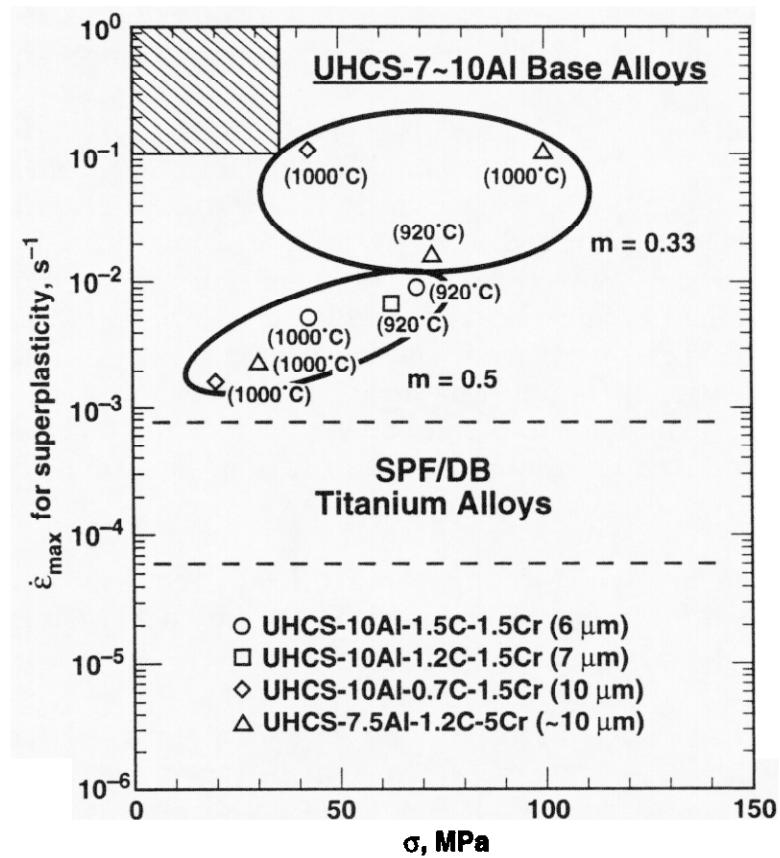


Figure 16. The maximum strain rate for superplastic flow, as a function of stress, for various UHCS-Al materials compared with the commercial goals set by the LLNL-industry consortium program (shaded area).

that the steel producer partner of the consortium could not make the UHCS-Al alloy by continuous casting because of breakout of the billet during casting; the aluminum created a brittle oxide skin which cracked during the bend portion of the vertical casting operation.

Room Temperature Properties

The original and principal focus of the development of UHCS was superplasticity. An early surprise, however, was the fact that the ultrafine structures developed for superplasticity were also highly effective in providing not only high room temperature strengths, but also excellent room temperature ductilities. Following this observation in 1975, it became immediately evident that the materials could have a powerful combination of high temperature formability and room temperature strength and ductility. Subsequent studies, therefore, inevitably investigated both these areas. Previous review papers [39, 73] have summarized various sets of these properties. The current range of available strengths and ductilities in an updated version of a UHCS is described next.

Examples of spheroidized microstructures and the corresponding room temperature stress-strain curves for a UHCS-1.8C-1.6Al material are shown in Fig. 17.

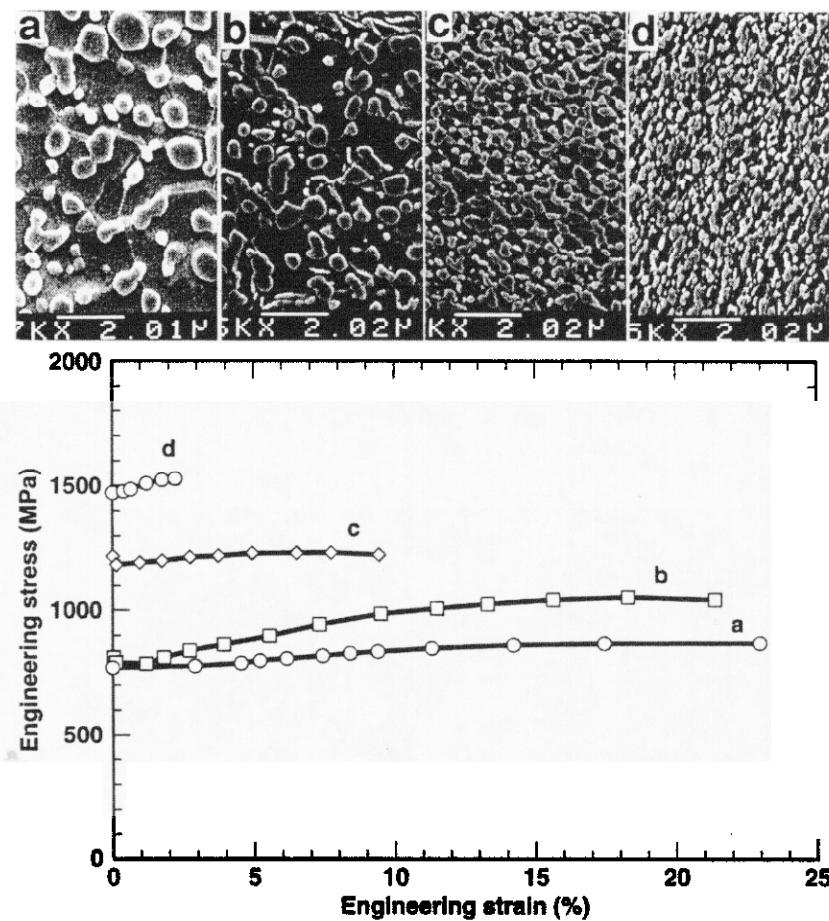


Figure 17. Scanning electron microscopy images and corresponding stress-strain curves for a UHCS-1.8C-1.6Al processed to yield various spheroidized carbide and ferrite grain sizes. The curve designations correspond to the micrographs.

Influence of Heat Treatment

The potential to heat treat UHCS compositions was recognized very early in their development. The unusual aspect of heat treatment of UHCS arose because after processing to develop an ultrafine structure, the steels could then be heat treated to achieve unusual microstructures and properties. For example unique martensitic, bainitic, and pearlitic structures could be developed and, furthermore, some of these structures exhibited compression ductility despite extremely high hardnesses and strengths [8, 39, 54].

Coarse and fine martensitic structures are illustrated in Fig. 18 from a UHCS-1.3C material. The structure of the UHCS material after water quenching from a high temperature of 1150°C is shown at the bottom right of Fig. 18. At

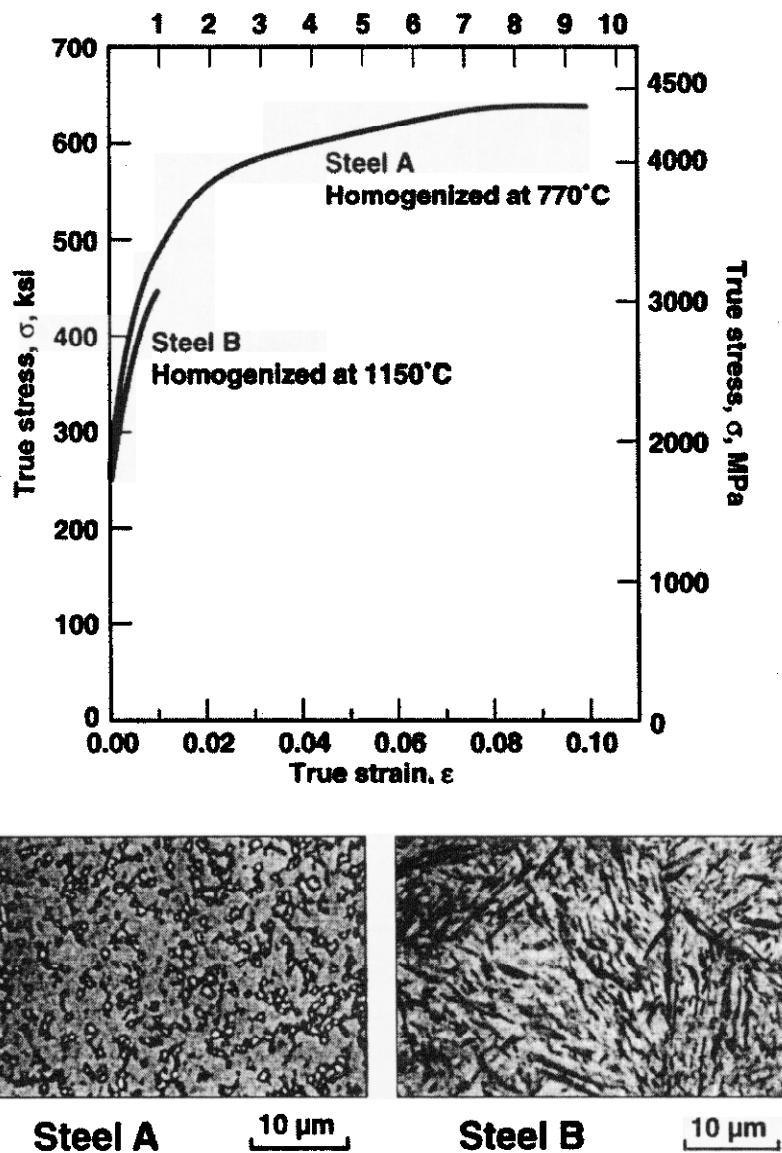


Figure 18. The influence of prior heat treatment on UHCS quenched from 770°C is shown above. On the graph, the compression stress-strain curves of a 1.3%C steel after two different prior heat treatments are shown with accompanying optical micrographs. The heat treatments involved, in one case heating to 770°C and quenching, and in the other case heating to 1150°C, cooling to 770°C, and then quenching.

this austenitizing temperature, most of the carbides are dissolved and the austenite grain size is coarse. Thus, the quenched structure is seen to consist of coarse martensite. Such a coarse martensitic UHCS material, although very hard, does not have any significant compression ductility at room temperature [8]. A quite different result is found if a fine-grained spheroidized structure is the starting point and the material is heated to a low austenitizing temperature (e.g., 770°C) and water quenched. In this case, the result is optically unresolvable martensite, having a nondescript appearance and a background consisting of submicron-size spherical (undissolved) proeutectoid carbides as shown in the lower left part of Fig. 18 for the UHCS-1.3C material.

This type of ultrafine martensite in a UHCS has unusual compression properties and the compression engineering stress-strain curves of a UHCS-1.25C steel and a number of tungsten-cobalt alloys are compared in Fig. 19. The fine martensitic UHCS is comparable to WC-12%Co in strength but has considerably greater compression ductility. The UHCS achieved an engineering strain in compression of 26% with an engineering fracture stress of 4690 MPa (680 ksi).

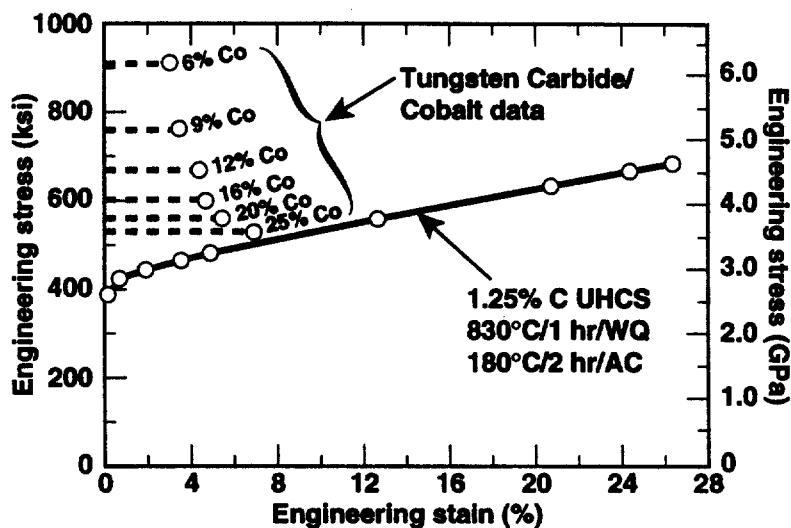


Figure 19. An engineering stress-strain curve for a fine-grained martensitic structure of UHCS-1.25C, which was tested in compression at room temperature. Comparison is made with a number of tungsten carbide-cobalt alloys.

The tensile properties of UHCS have also been evaluated for various heat-treated conditions: i.e., bainitic, tempered martensitic, and fine pearlitic. Examples are as follows. An ultimate tensile strength (UTS) of 1810 MPa (262 ksi) with 18% elongation was obtained for a fine bainitic structure in a UHCS-1.0C-1.5Cr material. A UTS of 1400 MPa (190 ksi) with 25% elongation was obtained for a fine pearlitic structure in a UHCS-1.25C-1.6Al material. When an ultrafine pearlitic structure was developed, a UTS of 1655 MPa (240 ksi) with 12% elongation was obtained for a UHCS-1.8C-1.6Al material [73].

A deficiency originally identified in unalloyed, ultrafine-grained UHCS was that of low hardenability. This is because grain boundaries are sources of nucleation of bainite and pearlite and, as such, inhibit the formation of martensite. It has been shown, however, that dilute alloying can enhance the hardenability of fine-grained UHCS [48, 73]. For example, the critical diameter is only 6.9 mm for a fine-grained plain carbon UHCS but can be increased to over 20 mm with dilute alloying additions of Mn, Cr, and Al.

UHCS Laminated Composites

In recent years, the concept of improved mechanical properties through the lamination of other materials to UHCS has been reviewed and investigated in some detail [12, 29, 54, 63, 91]. As mentioned previously, this class of materials also has a rich history involving the solid-state welding of strips of different steels or irons resulting in laminated steels having unusual surface markings and patterns. A number of famous "welded Damascus" products have resulted from these processes.

One of the attractions of developing laminates containing UHCS is that they can be solid-state diffusion-bonded readily, either to themselves or to other ferrous-base materials, at very low temperatures (below the A_1 temperature) because of their fine grains compared to other steels [12, 20]. This characteristic has led to composites with unique properties: at low temperatures, for example, they can have very high impact resistance [29]. At room temperature, lamination can lead to an improvement in the high-cycle fatigue behavior of UHCS [46] and has resulted in unique tensile characteristics [59, 71]. At intermediate temperatures, such laminated structures can exhibit superplasticity [33, 74].

In the area of impact resistance, high-impact strengths have been obtained in laminated composites. The first, and perhaps most dramatic example, was originally shown in 1983 for a UHCS/mild steel laminate [29]. The notch-impact strength of the laminated composite was shown to be high and exhibited a low ductile-brittle transition temperature of -140°C (much below that for either of the constituent components that make up the composite). The high impact resistance of the composite is attributed to notch blunting of the crack by delamination at the layer interfaces. The high impact resistance of the laminated composite in this case results from the presence of a good (but not perfect) bond between adjacent laminae. If the bond strength is improved, for example, by a heat treatment above the transformation temperature, the impact strength is degraded. In recent years the precise role of the interleaf materials, such as Hadfield Manganese steel (HMS) [56], Ni-Si steels [68], and brass [57, 61, 69], on the impact properties of UHCS has been studied in detail. The UHCS-brass laminate showed the highest impact resistance at low temperatures which was attributed to the presence of the notch-impact-ductile brass layers [69].

Tensile properties of laminated composites based on UHCS/304 stainless steel, UHCS/HMS, and UHCS/Fe-3Si alloy were evaluated [59] after various conditions of selective heat treatment. From these data, it was shown that the rule of averages correctly predicts the yield strength of the laminated

composites; however, the rule only predicts the ductility of the laminated composites when the ductilities of the two components are similar. In a subsequent study [71], laminated metal composites containing an equal volume percentage of UHCS and brass were prepared in three different layer thicknesses (750, 200, and 50 μm) and tensile tested at ambient temperature. A dramatic increase in tensile ductility (from 13 to 21 to 60%) and a decrease in delamination tendency at the UHCS-brass interfaces were observed as the layer thickness was decreased. This result is graphically illustrated in Fig. 20. The layer-thickness effect on ductility was attributed to residual stresses, the influence of which on delamination is decreased as the layer thickness is decreased. Suppression of delamination inhibits neck formation in the UHCS layers, resulting in extended uniform plasticity. In a related study [66], it was shown that crack propagation is made difficult in UHCS when it is interleaved with brass. An example is shown in Fig. 20 in which crack blunting and crack bridging events are seen to occur as a result of the presence of the brass layers.

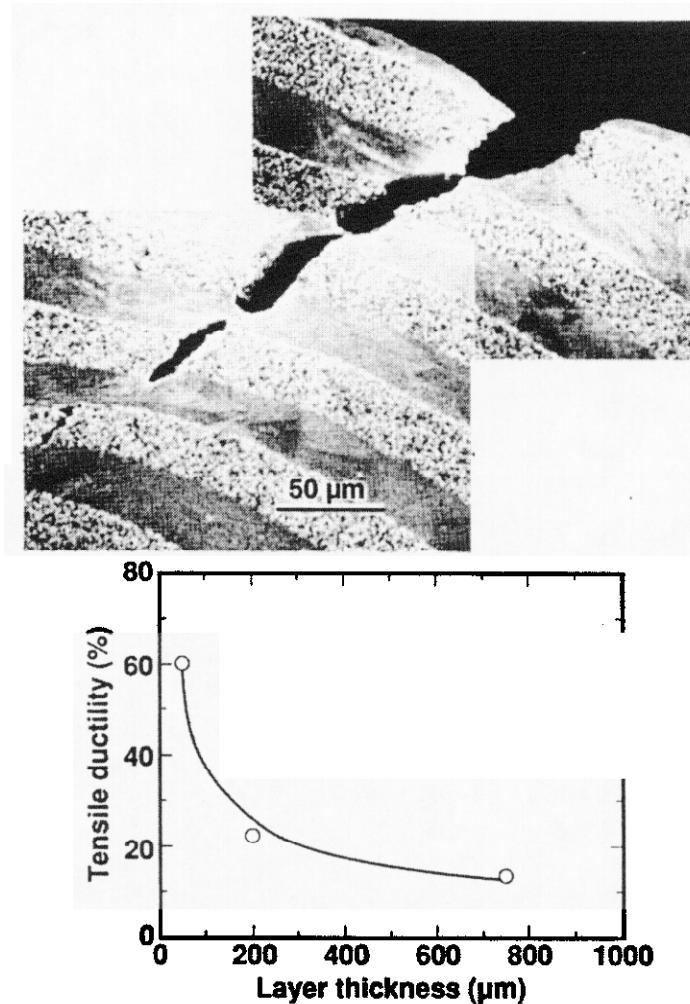


Figure 20. Tensile ductility as a function of layer thickness for UHCS/brass laminated composites. Tensile ductility increases with decreasing layer thickness because delamination is suppressed. Crack propagation in a UHCS/brass laminate is made difficult because of crack-blunting and crack-bridging events. The sample was tested by bending.

Ultrahigh carbon steel laminates consisting of superplastic and nonsuperplastic components have been shown to exhibit superplastic behavior [12, 44]. For example, UHCS/mild steel laminated composites have been shown to exhibit strain-rate-sensitivity exponents of over 0.30 and elongations to failure of over 400% [12]. It was shown, however, that high strain-rate sensitivity is a necessary but not sufficient condition for achieving superplasticity in UHCS laminates. When a UHCS/brass laminate is tested at elevated temperature, the brass layers fracture prematurely by grain boundary separation, leading to early failure of the composite. Recently [66], a laminated composite consisting of a ferritic stainless steel clad to a UHCS was predicted to exhibit ideal superplasticity at 800°C. Experimental studies demonstrated that this condition was achieved experimentally ($m=0.5$ and elongation to failure over 800%). This combination of components leads to the unexpected result that coarse-grained stainless steels can be made superplastic. Figure 21 illustrates the successful gas pressure forming of the superplastic UHCS/stainless steel laminate sheet contrasted with the failed stainless steel sheet which was not laminated. A detailed description of the superplastic behavior of UHCS-stainless steel laminates has been published [113].

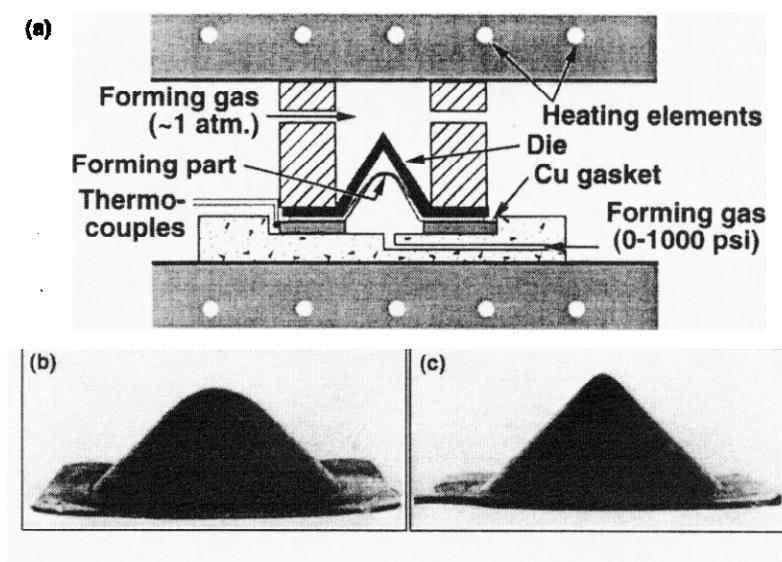


Figure 21. The gas-pressure forming of superplastic UHCS/stainless steel laminate sheet: (a) a diagram of the process, (b) a failed monolithic stainless steel sheet, and (c) the successfully formed laminate.

Potential Applications And Future Directions

Ultrahigh carbon steels have the following unique characteristics: (a) high hardness with compression toughness, (b) high strength with good tensile ductility, and (c) superplasticity. With these qualities, their potential for structural applications appears promising. The current applications that use high carbon steels (0.5 to 1.0% C) are logical candidates for substitution with UHCS. For example, eutectoid composition steels (0.8% C) are typically used for wires in tire reinforcement, cutting tools, and rails. The UHCS described here are a logical substitution in such applications because they have

ductilities comparable to the high carbon steels but with substantially higher strength, hardness, and wear resistance [73].

For forged or bulk rolled components, the primary characteristics of UHCS are wear resistance and their potential for net-shape processing (via superplasticity) to minimize machining and welding steps and the amount of scrap material produced. Ultrahigh carbon steels are excellent materials for wear resistant components in which high fracture toughness is not required. An added advantage of using a fine-grained UHCS is that the carburizing step normally required to harden the surface of components is eliminated.

For sheet or thin plate applications, high strength and good cold-stamping characteristics are often required. Ultrahigh carbon steels are a logical material substitution for high-strength sheet components that are strength limited rather than stiffness limited. The primary driver for high-strength steels in automotive sheet applications is often weight reduction which results in enhanced performance and fuel economy. There is a big drive to create "ultrahigh strength" sheet materials for automotive applications [87]. The considerable increase in strength of UHCS sheet is illustrated in Fig. 22 over conventional and advanced automotive steel sheet when such steels are compared at an equivalent tensile ductility.

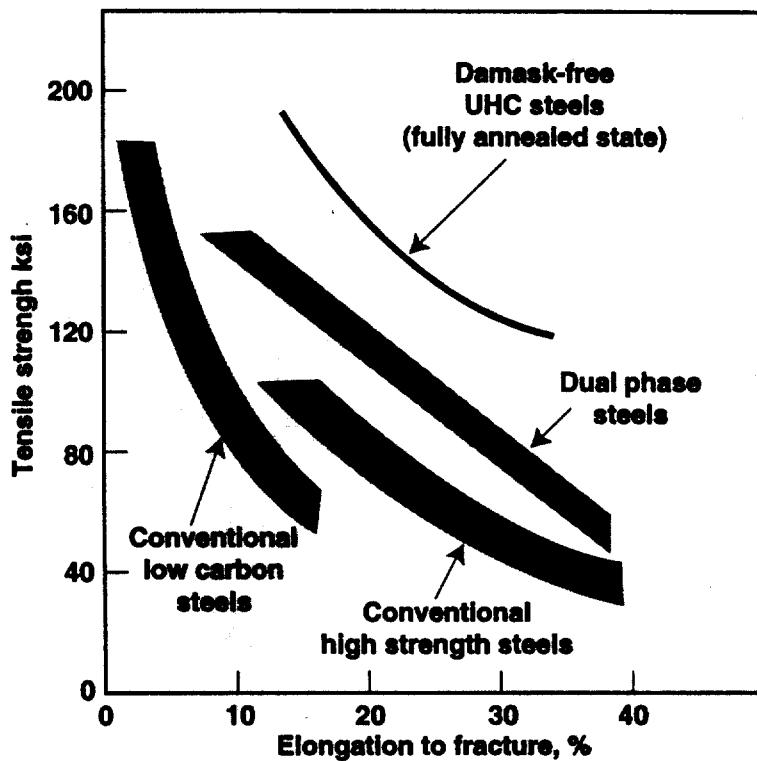


Figure 22. The considerable increase in strength of ultrahigh carbon steel sheet over conventional and advanced automotive steel sheet is illustrated when they are compared at an equivalent tensile ductility. The various strengths in the UHCS materials are achieved by different processing procedures and heat treatments ($\text{ksi} \times 6.895 = \text{MPa}$).

For wire or bar applications, the primary requirement is often strength. Ultrahigh carbon steels can be cold-rolled extensively at ambient temperature.

Thus, the cold-forming of UHCS into wire form is attractive. There is competition under way in developing stronger wires in eutectoid-composition-base steels for use in tires and in other applications. UHCS wires, with between 15 and 32 vol.% carbides, will lead to strengths that are higher than those obtained in wires with eutectoid-composition steels [88].

Two components made from UHCS were shown in Fig. 15. In addition, four other components made from UHCS are shown in Fig. 23. Each of the components was formed in one operation. The back-extruded tube and trunion were shaped at conventional forming rates ($\sim 10 \text{ s}^{-1}$) and illustrate the formability of fine-grained UHCS at high (non-superplastic) strain rates. The temperatures of forming were 750°C for the tube and 700°C for the trunion. The height reduction for the trunion was approximately 4 to 1. Both components were UHCS-1.25C-1.6Al. The guided missile aft-closure was forged at a temperature (815°C) and strain rate (10^{-3} s^{-1}) at which the UHCS-1.6C material exhibits superplasticity. The material for this component was fine-structure powder prepared via liquid atomization. The dome has a very uniform wall thickness illustrating uniform flow during forming. The subscale compressor hub was forged at near superplastic conditions (at 750°C and a strain rate of 10^{-3} s^{-1}) from a 40-mm-tall and 76-mm-diameter cylinder. This component, made from a UHCS-1.3C-1.6Al material, was formed as a part of a Department of Energy Metals Initiative Project at the Lawrence Livermore National Laboratory on superplastic steel processing. The component was formed to demonstrate the die-filling capability of UHCS by manufacturing a complex part to net shape.

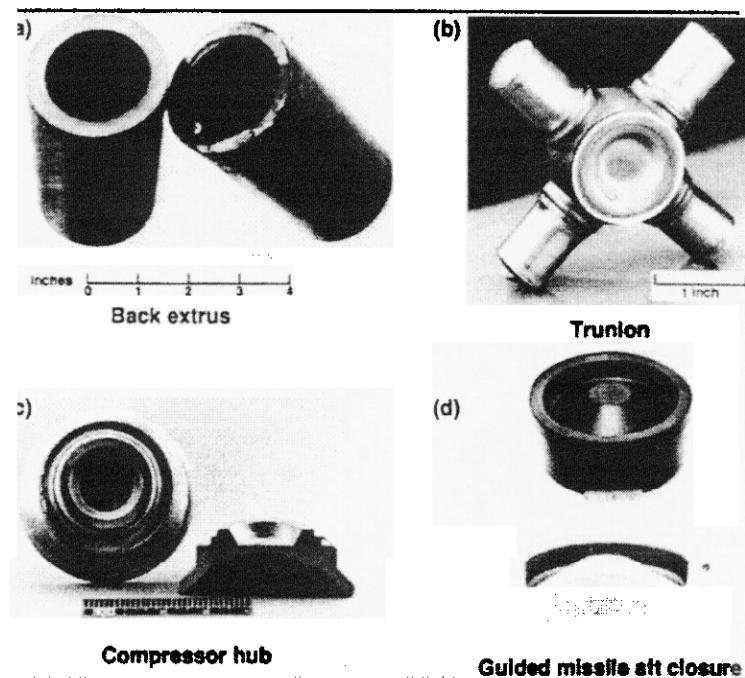


Figure 23. Components that have been formed from fine-grained spheroidized UHCS: (a) a back extrusion made at the Raychem Corporation (courtesy of Dieter Stöckel); (b) a trunion forged at the General Motors Technology center (courtesy of W. Mueller); (c) a subscale compressor hub from Pratt and Whitney (courtesy of Bryant Walker and Roy Athey); and (d) a forged guided missile aft-closure.

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