Ausferrite provides extreme strength and toughness for both ductile irons and steels!

My interest in ausferritic materials actually started at a tribology conference, Nordtrib 2006 in Elsinore, where Portuguese researchers showed that ausferritic ductile iron gears (ADI) are much more durable than steel gears against seizure!

I was then head of research at Indexator AB, which already had tribological challenges in both its hydraulic rotators and in the tiltrotator Rototilt, which is now the name of a separate company. The year before, we had started using what I (and later the rest of the world!) designate "Si-solution strengthened ferritic ductile iron". Shortly after the conference, I got the idea to investigate whether higher silicon levels are also beneficial for ADI, which led to Silléns Innovation Prize 2010 for the patented concept SiSSADI™. Thereafter the technology development company Ausferritic AB was founded, and since 2015 several patented and pending concepts for technology licensing have been developed, demonstrated in both SiSSASteel™ castings from Smålands Stålgjuteri and in rolled bar from Ovako. There is always something new to discover about ausferrite!

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PHOTO: AUSFERRITIC AB

Ausferritic ductile irons (ADI = Ausferritic Ductile Iron) and ausferritic steels are so far relatively unknown in Sweden, but regarding ADI ≈100 000 tons/year is used in the US and ≈10 000 tons/year in Europe. Within Sweden, Epiroc (previous part of Atlas Copco) Construction Tools in Kalmar are ADI pioneers, using it in very robust Hammer bodies for their Hydraulic hammers/breakers, and Epiroc Rock Drills has recently inaugurated an additional integrated salt bath facility in Örebro. Ausferritic steels are significantly less widely used globally, for various reasons described later in the article.

But what is ausferrite, and how does it work in ductile irons and steels?

Let's start by stating that steel consists of the element iron (Fe) with a *little* carbon (C), but if you have more carbon (>2 weight-% or >7.5 atom-%) the resulting metal is denoted <u>cast iron</u> where most of the carbon is, however, located in ≈10 volume-% of graphite as lamellas, spheroids or intermediate shapes, within a continuous matrix that can have a "steel-like" microstructure.

The technical terminology for microstructures in irons and steels that end with "-ite": *ferrite, austenite, pearlite, martensite, bainite, cementite* and now also *ausferrite*, describe atoms of mainly Fe + a little C and usually also other alloying elements in a certain crystal structure, i.e. in what regular ways the different atoms are located three-dimensionally at the nano level. The microstructures you get depend on the chosen combination of chemical composition and thermal history. Pearlite and bainite denote microstructures consisting of ferrite + an iron carbide known as cementite (Fe_3C). Bainite and ausferrite are the two youngest "-ite" concepts, from 1930´s and 1970´s respectively.

Ausferrite first appeared in ductile irons; it was actually described already in 1949 within the first ductile iron patent by Millis. However, since his alloys originated from grey irons having a *very high manganese* content, the transformation to his ausferrite was not *completed* resulting in *brittleness,* and *the use of ADI was therefore delayed for several decades!* The silicon content in ductile irons favors *grey* solidification, where the excess of carbon forms graphite following the stable phase diagram Fe−C instead of the metastable phase diagram Fe−Fe_3C commonly used for steels. *Silicon also favors the formation of ausferrite* if austenite is rapidly cooled to the intermediate temperature range between pearlite and martensite in a transformation diagram (TTT or CCT). In most steels, however, the Si content is too low for this to happen, resulting in the formation of bainite (fine ferrite + Fe_3C) instead.

In the steel field, bainite research has in Sweden mainly been carried out at KTH (by Hillert & Borgenstam) and internationally mainly at Cambridge (by Bhadeshia). *Ausferrite in high-silicon steels is, unfortunately, usually considered to be a special case of bainite* and is therefore often referred to as "superbainite", "nanobainite" or "carbide-free bainite", *although when the ferrite is formed in this case the carbon surplus stabilizes part of the austenite*, instead of forming Fe_3C in all kinds of bainite. The transformation into ausferrite takes place because an increased substitutional solid solution of Si atoms and/or Al atoms between the Fe atoms in the crystal lattice makes it much more difficult for Fe_3C to be formed.

In addition, the "Cambridge School" argues that the microstructure in all "bainite" first grows without diffusion (like martensite does) and that only then carbon completely (or partially) leaves the ferrite phase, while at KTH it is understood that the structure is formed under "paraequilibrium" when C-diffusion only (not larger atoms) occurs at low temperature.

In the case of *ordinary hardening* of steel, it is first heated yellow hot to transform into the austenitic crystal structure being stable at high temperature, so that significantly more C atoms can be interstitially dissolved between the Fe atoms than can be contained in ferrite (≤0.02 % C). Then the steel is rapidly cooled/quenched so that ferrite, pearlite and/or bainite do <u>not</u> have time to form during cooling. Characteristic of martensite is that when the M_S temperature of the steel is reached, the supercooled austenite begins to transform extremely rapidly into martensite with up to the speed of sound in steel (≈5000 m/s!). As a consequence, the carbon does not have time to be transported away by diffusion, resulting in a disordered ferritic crystal structure (Body Centered Tetragonal: BCT, not cubic BCC as in ferrite) as a result of the supersaturation with carbon.

The rapid transformation often leads to residual stresses and shape changes since rapidly cooling parts (thin sections and close to surfaces) transform into martensite earlier than slowly cooled parts (in the core of thick sections). The transformation is not completed until cooled also below the M_F temperature, and for steels with higher carbon contents having their M_F below room temperature, you sometimes have to resort to deep cooling to avoid residual austenite, which otherwise limits both maximum strength/hardness and dimensional stability.

The martensite is usually too brittle for use immediately after hardening, and therefore it is tempered at an intermediate temperature to transform it into a very fine microstructure of ferrite with low C content and into Fe_3C, both being much finer than in pearlite but also finer than in bainite because the initial formation temperature is lower. Steels hardened by tempered martensite are, therefore, both strong and reasonably tough, but the choice of tempering is always a compromise between strength and hardness on the one hand, as well as ductility and toughness on the other.

The <u>substantially new</u> with ausferrite having, like martensite and bainite, very fine microstructures on the nanolevel that restrict plastic deformation through dislocation movement and thus increase their strength, is that *in addition to the fine nanostructure, the strength of ausferrite is <u>not</u> based on iron carbides slowing down dislocations, but based on an unique <u>fully metallic</u> structure* in which mechanical interactions occur between a very tough but also strong <u>aus</u>tenite phase and a very strong but also tough <u>ferrite</u> phase, hence the concept of ausferrite.

How is the heat treatment carried out to obtain ausferritic microstructures?

The first two steps of the heat treatment for ausferrite (and for its "cousin" bainite) are reminiscent of those for common hardening to martensite, namely first an *austenitization* that is usually complete, but sometimes with a certain intentional percentage of unresolved pro-eutectoid ferrite in the austenitic matrix.

In the case of ductile irons, sufficient time is also needed to diffuse carbon from the graphite spheroids until reaching the solubility limit of carbon in the austenite, which is mainly depending on austenitization temperature and silicon content.

It is then followed by a *rapid cooling/quenching* so that new pro-eutectoid ferrite and/or pearlite do not have time to consume the austenite intended to be transformed into stronger microstructures. Similar to regular hardening, the material therefore needs to have sufficient *hardenability*, so for coarser wall thickness that cools more slowly, ductile irons are alloyed with copper and nickel as well as smaller amounts of molybdenum and manganese, while in steels copper is replaced with chromium and higher manganese levels can be allowed than in ductile irons.

It is relatively unknown that silicon also has a hardenability-enhancing effect at the same level as for nickel (but is much cheaper!), although this requires that the austenitization temperature is increased to compensate for the fact that the carbon solubility in austenite decreases with increased silicon content.

The increase in temperature may also be necessary for achieve complete austenitization since silicon, being a strong stabilizer of ferrite, shrinks the austenite field in the phase diagram, and the extent of this shrinkage has usually been underestimated (in both literature and simulation programs such as Thermo-Calc).

In relation to its relatively low content carbon in the austenite has, of course, the greatest impact on both how high the hardenability is and on how far down the M_S temperature is depressed.

What *is different from regular hardening,* however, is that quenching of the austenite takes place in a *salt bath*, i.e. <u>molten salt</u> (not salt water/brine!) that maintains the very temperature intended for trans-formation into ausferrite (or bainite). The choice of using a salt bath is based on the fact that although you want a rapid cooling, almost as fast as in hardening oils but slower than in water, you do not want to form martensite at even lower temperatures.

The left photo below shows the 70 L salt bath furnace donated in 2017 by Indexator Rotator Systems AB and Rototilt Group AB to Luleå University of Technology (LTU) for heat treatments of testing samples and single smaller components in research, while the right photo shows the charging of many larger samples in the industrial salt bath facility for Hammer bodies at Epiroc Construction Tools in Kalmar.

Salt bath oven (70 L) donated to Luleå University of Technology (left) and Integrated salt bath facility at Epiroc Construction Tools (right)

Inside an integrated salt bath facility, <u>two</u> *batches are simultaneously processed* – firstly above floor level in the austenitizing furnace with regulated carbon potential that can be seen in the middle of the photo (above the batch of vertical plates in the foreground), and secondly then transported via a gas-tight lock down to be quenched in the salt bath located below floor level (while the next batch is austenitized). The molten salt has about half the heat capacity per kilogram compared to cast irons and steels, so very large salt baths are used in order to limit the temperature rise in the salt to only a few degrees when the batch is quenched, and within 10-15 minutes cooled again to the intended temperature for the transformation into ausferrite. The salt is water-soluble, so in some industrial salt bath plants water can be continuously added to increase the quenching capacity of the bath, while in all cases the heat-treated components can be washed clean from salt residues with hot water, and after evaporation from the washing water the solid salt can be returned to the bath in a closed loop.

When the ductile iron or steel reaches the temperature of the salt bath, a usually *isothermal trans-formation* takes place during a large number of minutes, up to few hours. Therefore, *residual stresses and shape changes become modest*, but total process time for the entire heat treatment is *not longer*.

Like other phase transformations based on diffusion, such as pearlite, the transformation is completed in the shortest time at the best compromise between the *nucleation* of the ferrite (which benefits from high undercooling, i.e. low temperature) and the *growth* of the ferrite (which benefits from rapid carbon diffusion, i.e. high temperature). This "nose" at the shortest time in a TTT chart or a CCT chart is usually located at ≈350°C. Research at KTH has shown that the growth rate of the ferrite is often 1–10 μm/s, which is almost one million times slower than the growth rate for martensite (!), and thus fully compatible with the diffusion rate of carbon at actual temperatures.

Both the entire heat treatment and the third characteristic step is denoted *"austempering"* that literally means "tempering of austenite", being technical nonsense since austenite is the most ductile structure

possible in irons and steels, without any brittleness to temper! What is probably meant is that instead of cooling all the way down to room temperature (to form martensite) and then heating up again for tempering, you "temper" directly in the temperature range between pearlite and martensite.

How is ausferrite structured at the micro-/nano-level?

The **austenite phase** in transformed ausferritic microstructures consists mostly of thin (<<1 μm) films, which are _stabilized thermodynamically_ towards forming martensite by two factors: Partly by how thin the film is because transformation from densely packed austenite to less dense martensite results in a volume increase (which is more difficult to accommodate in a thin film surrounded by a growing ferrite phase), partly by its high carbon content at ≈1.5–2 weight-% C (≈3-5 times higher than in as-quenched austenite). The films are enriched by carbon diffusion from the majority of as-quenched austenite that is instead concurrently transformed into a very fine ferrite phase. This increase in carbon content causes the M_S-temperature of the austenite phase to drop by several hundred degrees and in many cases, it is thermodynamically stable against forming martensite even when immersed in liquid nitrogen at −196°C!

The **ferrite phase** in ausferritic microstructures consists of packages of thin (<<1 μm) plates, that often look like thin needles in microscopy cross-sections. This ferrite is very strong due to its supersaturation with interstitially dissolved carbon (since the formation of cementite is avoided due to the high silicon content and the remaining austenite minority cannot dissolve all carbon!), resulting in a disordered ferritic crystal structure (BCT) similar to untempered martensite. In addition, the high silicon content contributes to a substitutional solid-solution strengthening effect in the ferrite phase, with ≈80-100 MPa per weight-% Si.

However, the high-carbon austenite phase is _not_ _mechanically_ stabilized against locally forming deformation martensite in case of severe plastic deformation. Then the material swells locally and thereby "strangles cracks", resulting in an _elasto-plastic fracture toughness K_{JIC} being unusually high for very strong materials_. Concurrently, extensive strain hardening can provide high ductility through a gradual and partial transformation of the austenite phase into deformation-induced martensite. In addition, this mechanism results in high surface hardness if shot peened and in gear contacts subject to high Hertzian stresses, as well as a very good resistance to abrasion.

Paramount for ausferrite is that its austenite phase is **"sufficiently" stable!**

In the finished ausferritic microstructure the austenite phase is in minority relative to the ferrite phase, and the proportion of this _intended_ austenite (which thus does not have much in common with the unwanted and harmful "residual austenite" after martensitic hardening!) _is mainly determined by the salt bath temperature T_{AF}_ at _which ausferrite is formed_.

The closer T_{AF} is just above the $M_{s\,initial}$ temperature, where the as-quenched austenite would have started to form martensite if cooling would have continued instead of stabilizing at the salt bath temperature, the smaller the proportion of austenite phase and the finer the micro-/nano-structure becomes in the finished ausferrite.

If T_{AF} is instead far above the $M_{s\,initial}$ temperature, the microstructure becomes less fine and with a larger proportion of austenite phase.

Greatly simplified, ausferrite formed at a lower T_{AF} results in a finer structure (due to rapid nucleation but slow growth) with more ferrite phase and thus a higher strength and hardness, while ausferrite formed at a higher T_{AF} gets a less fine structure (due to slow nucleation but rapid growth) with more austenite phase and thus potential for a higher ductility _but in this case, there are pitfalls_; more about this later.

An example of the effect from five (5) different salt bath temperatures T_{AF} on the mechanical properties of both one continuously cast ductile iron bar (thus without any macro-porosity) according to the SiSSADI™ concept and one forged steel according to the SiSSASteel™ concept, where both materials had similarly high silicon levels (>3 weight-% Si) and medium carbon levels (≈0.5 weight-% C in the steel, as well as in the ductile iron matrix after austenitization but before quench), are shown by the resulting ten (5×2) tensile testing curves and corresponding mechanical properties in the following graph:

Spänning [MPa]

*Liknande Si% & C% i ausferritiskt stål (solida linjer)
och i ADI-matrisen (prickade linjer);
identisk saltbadstemperatur för varje färg*

SiSSASteel Cr04 (555 HBW): $R_{p0.2}$=1384 MPa; R_m=1885 MPa; A_5=13.3% (saltbad vid lägsta T_{AF})
SiSSASteel Cr06 (534 HBW): $R_{p0.2}$=1309 MPa; R_m=1779 MPa; A_5=13.7% (saltbath vid låg T_{AF})
SiSSASteel Cr09 (514 HBW): $R_{p0.2}$=1212MPa; R_m=1640 MPa; A_5=18.1% (saltbad vid medelhög T_{AF})
SiSSASteel Cr13 (477 HBW): $R_{p0.2}$=1032 MPa; R_m=1565 MPa; A_5=20.9% (saltbad vid hög T_{AF})
SiSSASteel Cr14 (401 HBW): $R_{p0.2}$=701 MPa; R_m=1617 MPa; A_5=20.0% (saltbad vid högsta T_{AF})

SiSSADI CF15 (495 HBW): $R_{p0.2}$=1352 MPa; R_m=1679 MPa; A_5=3.2% (saltbad vid lägsta T_{AF})
SiSSADI CF10 (477 HBW): $R_{p0.2}$=1288 MPa; R_m=1591 MPa; A_5=3.0% (saltbad vid låg T_{AF})
SiSSADI CF09 (429 HBW): $R_{p0.2}$=1008 MPa; R_m=1430 MPa; A_5=6.5% (saltbad vid medelhög T_{AF})
SiSSADI CF04 (388 HBW): $R_{p0.2}$=970 MPa; R_m=1300 MPa; A_5=10.7% (saltbad vid hög T_{AF})
SiSSADI CF01 (352 HBW): $R_{p0.2}$=842 MPa; R_m=1150 MPa; A_5=11.4% (saltbad vid högsta T_{AF})

Töjning [%]

Tensile testing curves and corresponding mechanical properties of one ausferritic steel (solid curves) and one ausferritic ductile iron (ADI, dotted curves) having similarly high Si levels and medium C levels, austempered at the same color-coded five (5) salt bath temperatures, where the range is 110 K between coldest (violet) and warmest (red) salt bath temperature

At the lower end of the temperature spectrum, we have the steel represented by a solid violet curve and the ductile iron represented by a dotted violet curve, which both transformed into ausferrite at the same lowest T_{AF}, in this case only 10 K above the $M_{s\,initial}$ temperature of their as-quenched austenite. This resulted in *very fine microstructures with only ≈5 volume-% austenite phase and thus very high strengths and hardnesses*.

At the higher end of the temperature spectrum, we have the steel represented by a solid red curve and the ductile iron represented by a dotted red curve, which both transformed into ausferrite at the same highest T_{AF}, in this case as much as 120 K above the $M_{s\,initial}$ temperature of their as-quenched austenite. This resulted in *less fine microstructures with ≈40 volume-% austenite phase and thus higher ductility, defined as the elongation at fracture A_5*.

However, it should be noted that the two orange curves with ≈30 volume-% austenite phase actually represent *more useful material properties*. This is because red materials have so much austenite phase that on the one hand, their proof strengths $R_{p0.2}$ and thus their usable strengths are relatively low, while on the other hand, there has not been enough carbon available to increase the carbon content of the intended austenite phase sufficiently to optimally delay gradual and partial transformation of the austenite phase into deformation-induced martensite, which could have resulted in an even higher ductility.

Despite the high ultimate tensile strength R_m=1779 MPa for SiSSASteel™ "CR06" (blue solid curve & data) it actually *set a record in elasto-plastic fracture toughness*: K_{JIC}=152 MPa√m. This means a great ability to carry unevenly distributed stress levels, so that up to a macroscopic stress level corresponding to its proof strength $R_{p0.2}$=1309 MPa, the steel will plasticize and redistribute the load around an atomically sharp crack instead of immediate fracture, as long as the crack length does not exceed $a_{crit.}$ =13.5 mm!

This **"robustness"** is on the same level as for the well-known tempered martensite steel 34CrNiMo6 Q+T (or AISI 4340), but *both proof strength and fracture toughness of the ausferritic steel is 50% higher!*

When you temper the martensite at a lower temperature to get comparable proof strengths, it becomes significantly less "robust", since its critical crack length for immediate fracture reduces to $a_{crit.}$ =2.3 mm.

An overall observation in the graph is that all ausferritic materials *deformation harden significantly*, but that the ausferritic ductile irons (ADI´s) do not form significant necking before fracture, as a result of the graphite spheroids counteracting this. It can also be noted that HBW hardness and ultimate tensile strength R_m of the three strongest ausferritic ductile irons are approximately 90% of the values of the

three strongest ausferritic steels, austempered at the same T_{AF}. This may be due to the fact that ADI have only ≈90 volume-% of ausferritic "steel-like" matrix, while the rest consist of graphite spheroids.

The SiSSADI™ (Silicon Solution Strengthened Ausferritic Ductile Iron) concept, patented by Ausferritic AB, has a more powerful solid solution strengthening of the ferrite phase by at least 3.35 weight-% Si. It offers *several advantages when you want a relatively ductile ADI with high ultimate tensile strength levels*, from 1200 MPa and upwards, because even at the low T_{AF} used for austempering just above the $M_{s\ initial}$ temperature the precipitation of embrittling Fe_3C can be avoided, something that in conventional ADI with ≈2.4 weight-% Si is only possible when austempering is carried out at higher T_{AF}.

On the other hand, conventional ADI is *more suitable for the remaining EN & ISO-standardized lower ultimate strength levels at 1050 MPa and 900 MPa*, because lower Si contents provide higher C solubilities during austenitization, and thus there is more carbon available to stabilize larger austenite phase proportions in these grades.

What has happened recently for ausferritic steels to catch up with ADI?

The Swedish project "AusFerrit" during 2016-2019 with seven industrial participants as well as LTU, co-financed by VINNOVA (Swedish Agency for Innovation Systems) via the Strategic Innovation Program "Metallic Materials" and being coordinated by Jernkontoret (Swedish Steel Producers Association), was **focused on ausferritic steels**, since these are significantly less developed than ADI but **with an even greater development potential, since the steels can also be forged and rolled**. During this project, the production processes were significantly scaled up for steel alloys having medium carbon and high silicon contents, being suitable for heat treatments into ausferrite.

Before the start of the project, a few small *forged lab ingots* (80 kg) had been produced in collaboration between Ausferritic AB and Uddeholm AB (a world-leading manufacturer of tool steels for industrial tools).

Through a process engineering bridging phase during the first two years of the project, several relatively small *1-tonne batches* of SiSSASteel™ alloys "G2" and "G4" were *first cast as rolling ingots*, each using the entire furnace capacity at Smålands Stålgjuteri AB (SSG, a medium-sized steel castings foundry in Eksjö), *followed by forging into a single rolling billet* with cross-section 160×160 mm at Ovako (a leading European manufacturer of engineering steels) in Hofors, and *finally by hot rolling to flat bars or round bars* at Ovako in Smedjebacken.

During the third and final year of the project, a *full continuous cast batch* of SiSSASteel™ alloy "G6" was produced by Ovako in Smedjebacken *into just over 100 tonnes of rolling billets*, which on three occasions so far has been *hot rolled into various bar geometries* in Smedjebacken and in Boxholm.

In addition, *steel castings* of SiSSASteel™ alloy "G3" were cast at SSG in Eksjö within the project.

Photos below show, from left to right, steel bars based on forged ingot "G2" rolled to flat bar 200×10 mm, steel castings "Grating teeth" cast in alloy "G3" after austempering heat treatment at Epiroc, steel bars based on forged ingot "G4" rolled to round bar Ø53 mm, and bar steel based on continuous cast billets "G6" which in this case was rolled to rectangular spring steel profile 95×49 mm with rounded corners.

Four examples of steel produced in project "AusFerrit": Ingot rolled to flat bar 200×10 mm, steel castings "Grating teeth" after austempering heat treatment, ingot rolled to round bar Ø53 mm, and continuous cast billets rolled to spring steel profile 95×49 mm

The most revolutionary discovery during the project was that the microstructure in rolled Ø53 mm bars made from SiSSASteel™ alloy "G4" was _not_ perlitic but instead **_became predominantly ausferritic during cooling after hot rolling_**, and in addition that the **_ausferritic structure established during cooling can be completely transformed by "baking" in air._**

The steel was apparently sufficiently alloyed to avoid forming pearlite during air cooling of Ø53 mm bars with ≈30 K/min in their core on the cooling bed after hot rolling.

It was **_unexpected that ausferrite would form to such an extent during the even slower cooling by ≈5-10 K/min_** after shearing into 6-meter bar lengths at 460°C **_during their continued cooling to 320°C in ≈10 minutes_**. The transformation into ausferrite is considerably faster in steels than in ductile irons!

However, _the ductility in "as-rolled" condition was not good_, due to the fact that in the predominantly ausferritic microstructure there were _"islands" consisting of not completely transformed austenite_.

Although the austenite islands were sufficiently C-enriched to be stable against forming martensite thermally during cooling to room temperature, they were **_extremely unstable mechanically_**, which on the one hand resulted in a too early and extensive transformation into deformation-induced martensite resulting in fracture far before necking and thus limiting both ultimate tensile strength R_m and elongation at fracture A_5, and on the other hand (before that) resulted in a low proof strength $R_{p0.2}$ because thicker austenite plasticizes more easily.

My research colleague Per Rubin in Luleå then suggested that **_the predominantly ausferritic microstructure established during continuous cooling may be "baked" in air to complete the transformation_**.

Fortunately, we had two extra tensile testing bars available, so these were "baked" in Per's kitchen oven!

Changes in microstructure and hardness did not become so great after "baking" except that **_the austenitic "islands" disappeared_** (the austenite phase appear bright in light optical microscopy after etching with Nital):
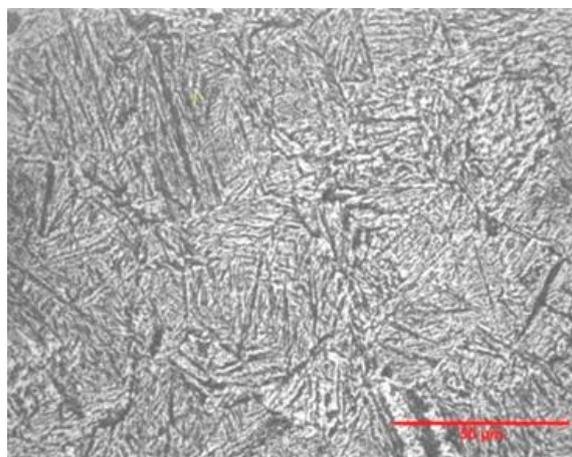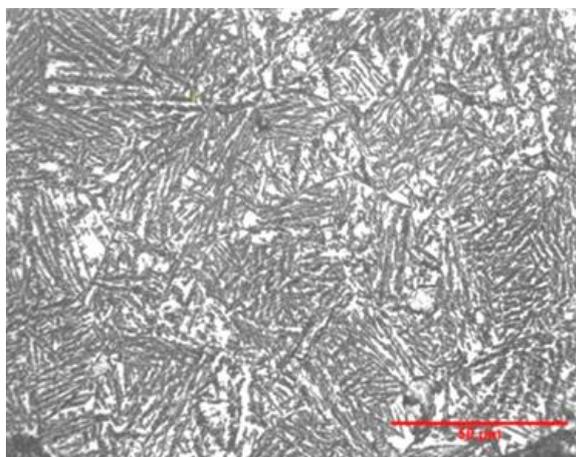
Microstructures with 90% ausferrite established during air cooling after hot rolling to Ø53 mm bar (left) and transformation to ausferrite completed after baking (right); hardness levels were 415 ±15 HV30 and 431 ±4 HV30 respectively; light optic microscopy (LOM) after etching with Nital: red line corresponds to 50 µm

However, the ***differences in mechanical properties and resulting fracture surfaces became dramatic:***
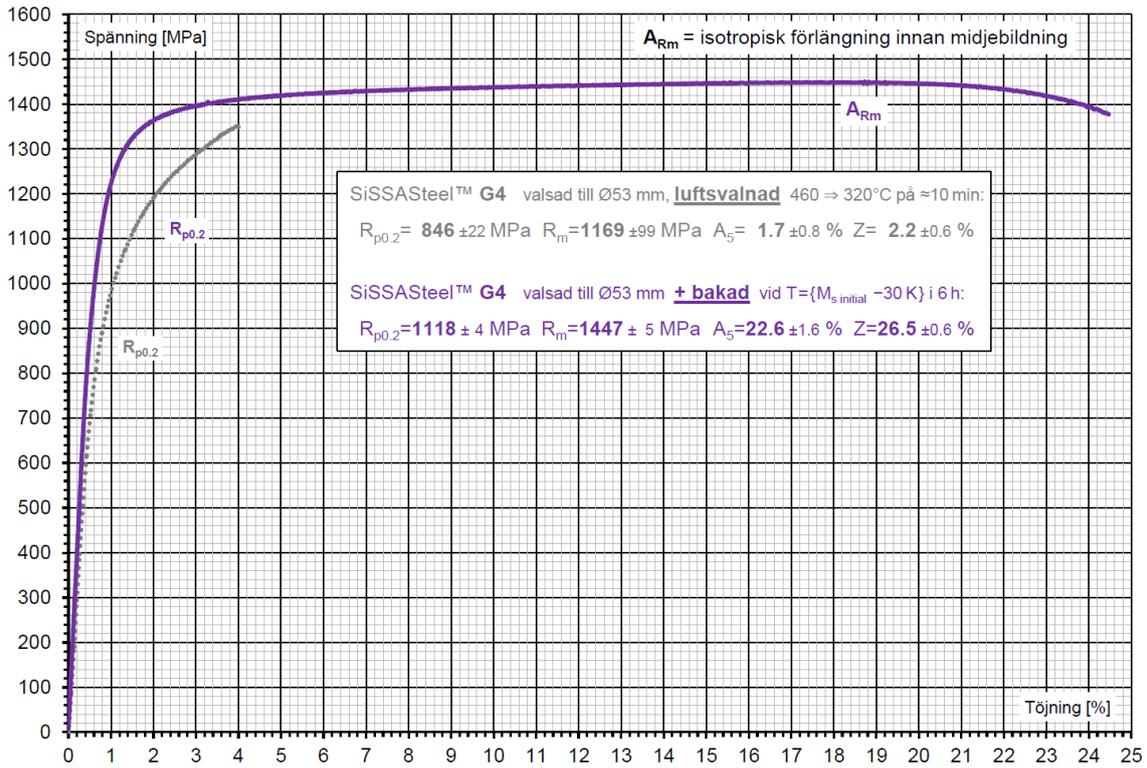
Tensile testing curves and corresponding mechanical properties for ausferritic steel SiSSASteel™ G4, after hot rolling to round bar Ø53 mm followed by air cooling (grey dotted curve), vs. after an additional baking in air at low temperature (violet solid curve)
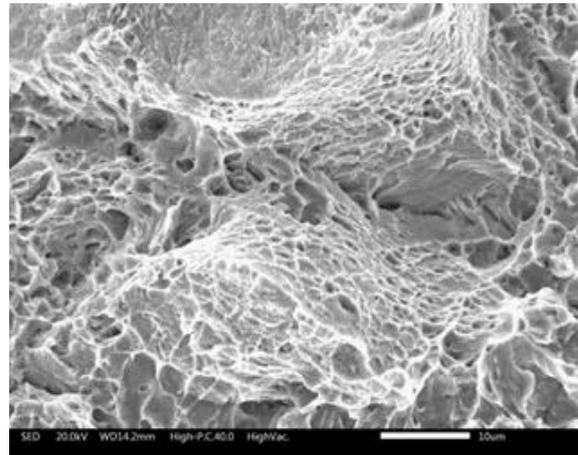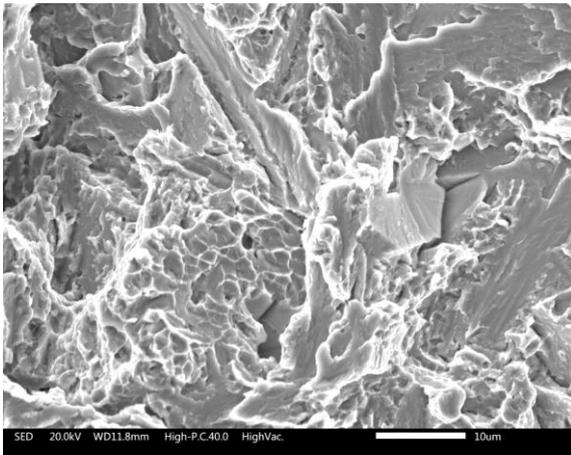
Fracture surfaces in ≈90% ausferrite established after hot rolling to Ø53 mm and air cooling (left), vs. completed 100% after an additional baking (right); in the air-cooled only fracture surface on the left, ductile fracture (dimples) are visible in the left part of its image but also in the right part smaller areas with brittle fractures (cleavage) are found, probably corresponding to mechanically unstable austenite islands, while the baked fractured surface in the right image is completely ductile (extensive dimples only); scanning electron microscopy (SEM): white line equals 10 μm

Continued baking experimentation has shown that similarly beneficial results can be obtained throughout the temperature range that would result in isothermal ausferrite (using conventional austempering in salt bath), provided that combinations of temperature and time during baking are selected so that comparable carbon diffusion is allowed to occur.

Baking has also been shown to produce very positive effects <u>after</u> conventional austempering (carried out by separate austenitization followed by rapid cooling and isothermal transformation in salt baths), although in those cases any clear change in the microstructure can rarely be seen by LOM. For example, below are mechanical properties for the continuous cast and hot rolled SiSSASteel™ "G6" steel, austempered at a *medium* salt bath temperature (held at 40 K above the $M_{s\ initial}$ temperature of its as-quenched austenite):

SiSSASteel™ G6 V, austempered only:

$R_{p0.2}$ = **1041** ±33 **MPa** R_m = **1766** ±16 **MPa**

A_5 = **14.8** ±1.2 **%**   Z = **39** ±6 **%**

Fracture toughness K_{JIC} = **96.0** ±0.8 **MPa√m** ⇒ $a_{crit.}$ = 8.5 mm

KV_L = **29.9** ±2.4 **J** KV_T = **23.7** ±0.6 **J** $KV_{L-20°C}$ = **17.3** ±2.5 **J**

SiSSASteel™ G6 X, austempered + baked:

$R_{p0.2}$ = **1212** ±4 **MPa** R_m = **1710** ±2 **MPa**

A_5 = **15.6** ±0.9 **%**   Z = **48** ±0 **%**

Fracture toughness K_{JIC} = **115** ±6 **MPa√m** ⇒ $a_{crit.}$ = 9.0 mm

KV_L = **31.6** ±2.9 **J** KV_T = **27.0** ±0.6 **J** $KV_{L-20°C}$ = **20.0** ±1.8 **J**

The improvements that show that the baking completes the transformation into ausferrite (when necessary) *and also equalizes the carbon content within the austenite phase* (which is always beneficial) *are,* on the one hand, ***significantly increased proof strength $R_{p0.2}$***, and on the other hand ***significantly less variations in mechanical properties*** between the three tensile test bars in the baked case.

The range of microstructures and mechanical properties that can be obtained for continuous cast and hot rolled SiSSASteel™ "G6" ***after austempering in salt baths at different T_{AF}*** (being either 30 K higher or 30 K lower than above), in both cases followed by the same baking in air, is shown in the following microstructures and graph:
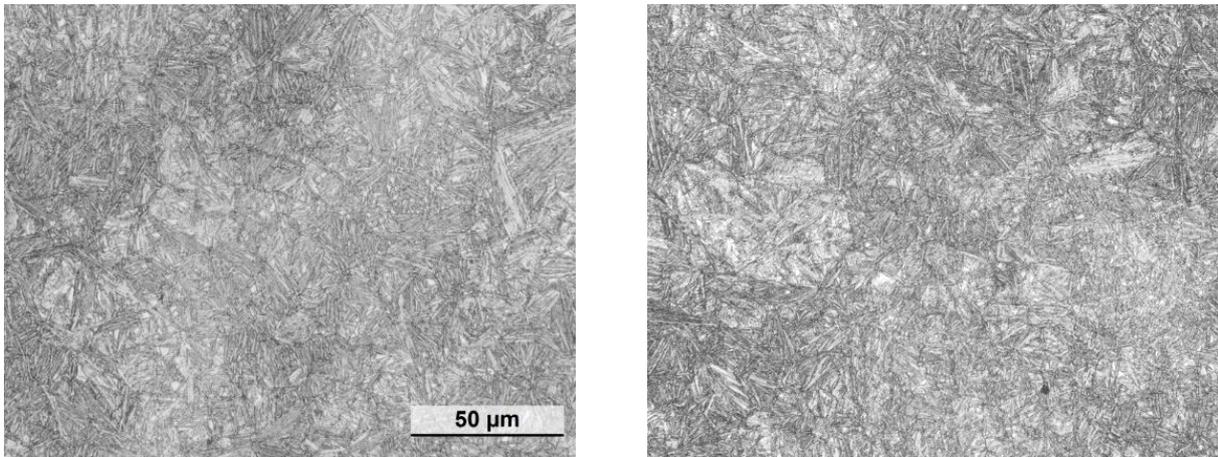
Microstructures with ausferritic steel SiSSASteel™ G6, after austempering at lowest salt bath temperature (left, blue curve in following graph) vs. highest salt bath temperature (right, red curve in following graph), in both cases followed by the same baking in air; light optical microscopy (LOM) after etching with Nital: black line equals 50 μm
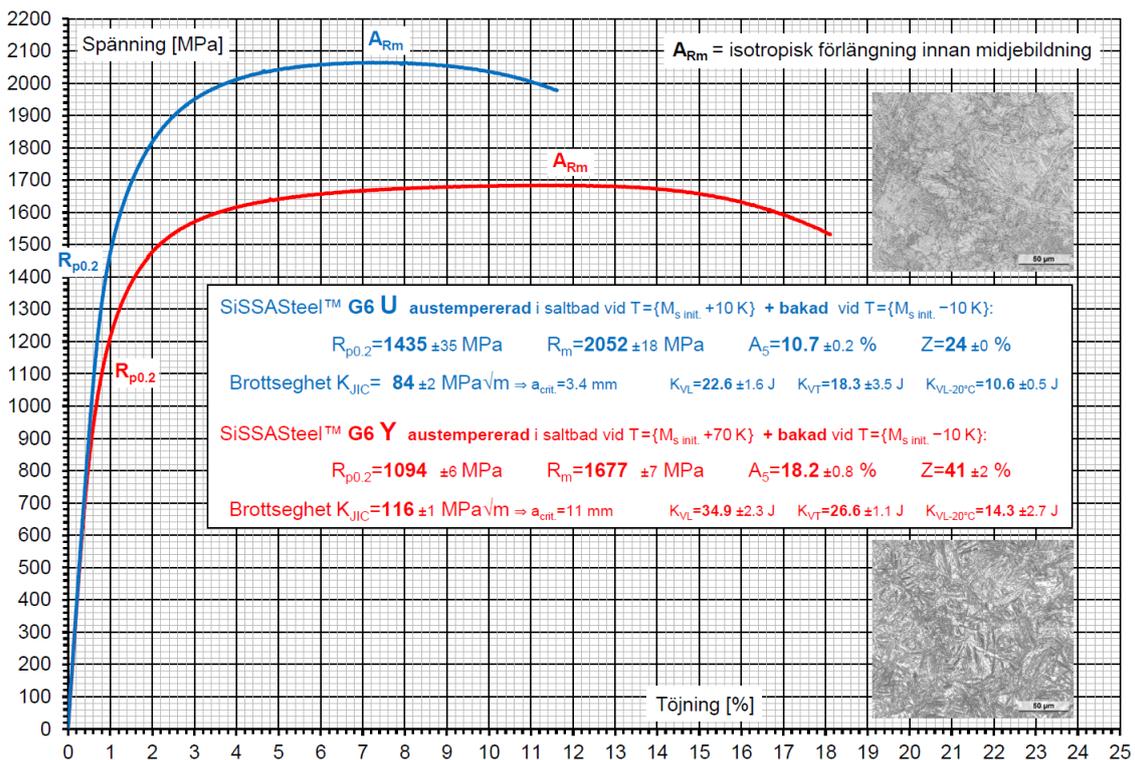
Tensile testing curves showing the range in corresponding mechanical properties for ausferritic steel SiSSASteel™ G6, after austempering at the lowest (blue curve) or at the highest (red curve) salt bath temperature, in both cases followed by the same baking in air

Steels concurrently providing an ultimate tensile strength R_m above 2000 MPa and a fracture elongation A_5 in double digit percent is very rare, and such aerospace steels cost significantly more in both alloying and production process! An additional advantage is that ausferrite, due to its continuous austenite phase, is significantly less sensitive to hydrogen embrittlement when compared with high strength martensite.

Summary of results

The project demonstrated that it is technically possible by several different processes to produce steels suitable for transformation into ausferrite (in alloys characterized by high Si and medium C contents), both as shaped steel castings and as hot rolled steel bars from forged ingots or continuous cast rolling billets.

The ausferritic structure can be formed both faster and in more ways in steels, either like ductile irons by conventional austenitization followed by rapid cooling and isothermal transformation in salt baths, or established during continuous slow cooling of steel provided that pearlite is not previously formed.

For ausferritic structures rapidly being established and becoming thermally stable in steels, but where remaining "islands" of untransformed austenite are not mechanically stable enough against forming martensite early during plastic deformation, the transformation must be completed by *"baking"*, but such baking can take place at low temperature in air and using slow changes in temperature (since the austenite may neither transform into pearlite, nor into martensite).

During the baking process step, as well as during the previous establishment of predominantly ausferrite (either isothermally or during slow cooling), higher levels of *silicon contribute to an expanded process window,* by accelerating the transformation of austenite into ausferrite (since Si is a strong ferrite stabilizer), while reducing the kinetics of possible degradation of the high-carbon austenite phase of ausferrite into bainite and/or the precipitation of bainitic carbides from its supersaturated ferrite phase (in spite of both processes being thermodynamically favorable). The reduced kinetics for degradation processes in the ausferrite by increased Si may also extend applications for ausferrite to continuous use at slightly elevated temperatures, or momentarily to even higher temperatures.

The establishing of ausferrite must have proceeded so far that most of the austenite has been transformed into ausferrite, and not more than a small amount of martensite has formed. If the latter has happened, such martensite may be tempered at low temperature during baking of the ausferrite and provide additional strength, as the elevated Si content in addition to its other merits also prevents temper embrittlement of martensite. This is already used in 300M-type aerospace steels where the designation indicates that the martensite is tempered at 300°C (without embrittlement due to the elevated Si content).

When the ausferrite is established during continuous cooling, it begins to form already at higher temperatures and initially becomes coarser with a higher proportion of C-stabilized austenite. The strength cannot, therefore, be as high as for finer ausferrite structures having a much greater proportion of solution-strengthened ferrite, when formed during isothermal austempering in salt baths at lower temperatures.

During baking, a new generation of ausferrite is formed from the "austenite islands", with fineness and austenite−ferrite proportions mainly determined by the baking temperature. Since the new ausferrite represents a small volumetric proportion and is not continuous, the ultimate tensile strength R_m is usually not affected much by baking, except in similar cases as the mechanically unstable "austenite islands" in continuously cooled SiSSASteel™ G4. However, both proof strength $R_{p0.2}$ and the ductility defined as the elongation at fracture A_5 can often be significantly improved, while minimizing the variation in properties.

Note that unlike the isothermal transformation in salt baths of as-quenched austenite, baking can *occur at a lower temperature* than the $M_{s\,initial}$ temperature that the austenite had before any ausferrite was formed, since the increased carbon content in the austenite greatly reduced its M_s and made it thermally stable. However, the transformation during baking also depends on carbon diffusion, so the lower the selected temperature and diffusion distances, the longer the completion takes.

It could also be emphasized that for rolled (or forged) steel cross-sections that cool within a suitable range (neither too slow resulting in pearlite, nor to rapid resulting in martensite), *the continuous cooling + baking route resulting in ausferrite has the potential to offer considerably reduced heat treatment costs*, compared with the conventional quenching and tempering process for martensite.

Low temperature air baking and flat temperature ramps *can also be used to **shorten time-consuming salt bath cycles*** in integrated salt bath facilities, *in this case **for both ductile irons and steels***. To ensure that the transformation to ausferrite has been completed, the holding time of the batch in the salt bath is usually significantly longer than the time that the batch needed to be in the previous austenitization furnace. A subsequent and inexpensive baking step (such as in a continuous belt oven) *allows both holding times to be similarly short, increasing the productivity* of such a relatively expensive equipment and *lowering the total process cost*, concurrently with *making the material properties even more robust*!

Both the SiSSASteel™ concept, the concept for ausferrite after continuous cooling of steel followed by baking and the concepts for shortened salt bath treatment of ductile irons and steels followed by baking are patent pending by Ausferritic AB and open for technology licensing.

FACT BOX

Dr. Richard Larker was presented in depth in Swedish journal "Gjuteriet" #5 2020, but here is an update:

Richard is currently on leave of absence as CTO at Ausferritic AB but still has the company office at Rototilt Group AB at Vindeln Innovation Park, and is since December 2020 employed as senior lecturer in Mechanical Engineering at Umeå University. There he teaches Engineering Materials and continues within 40 % of the appointment his research on ausferrite, among other things with UmU's special SEM where phase transformations during baking at elevated temperature can be studied "in-situ". He has also since 2019 continued to be an adjunct professor in Materials Engineering at LTU, specialising in high strength steels, and has identified synergies with a deepened collaboration in north of Sweden within both material research and teaching.

If interested, please take contact by e-mail: Richard.Larker@rototilt.com or Richard.Larker@umu.se