THE EFFECT OF AGEING ON THE SPOT WELD STRENGTH OF AHSS AND THE CONSEQUENCES FOR TESTING PROCEDURES

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ABSTRACT

Market trends within the automotive industry are leading to an ever-increasing use of high-strength and advanced high-strength steels (AHSS). The attraction of these materials is the advantage of excellent formability, combined with increasingly high tensile strength. It is a well-known fact that weld performance can be an issue with AHSS, where susceptible weld microstructures can lead to low strengths and undesirable failure modes. Much research has been conducted and published on this subject. A less well-documented effect in the weld performance of AHSS is 'ageing', whereby a weld exhibits poor mechanical performance immediately after welding, but after a certain period of time, the weld properties improve significantly. In the Corus – SMI research cooperation, this 'ageing' effect was first observed in weld samples in 2004, since this time ageing has been a major topic of combined research. This presentation is a summary of the Corus – SMI weld ageing study, highlighting the major issues and characteristics of the effect: the influence of process parameters, the susceptible alloying systems and the possible mechanisms that can cause ageing of the weld. The 'ageing' effect has serious implications for standardized testing procedures, where the timescale between welding and testing is not specified, ageing can have a huge influence on the welding results obtained in ageing susceptible materials. The final aspect of this report is to consider the consequences of ageing for weld testing procedures.

IIW-Thesaurus keywords: Ageing; Austenite; Automobile engineering; Brittle fracture; Cruciform joints; Ductile fracture; Fractures; Gases; High strength steels; Hydrogen; Martensite; Mechanical properties; Peel strength; Reference lists; Resistance spot welding; Resistance welding; Steels; Strength.

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1 INTRODUCTION

In recent years, two trends have been dominating design and manufacturing in the automotive industry. On the one hand, there is a trend to reduce the weight of vehicles, driven by the desire to decrease fuel consumption and the release of greenhouse gases. On the other hand, there is a trend to improve the structural rigidity of vehicles (especially the crash performance), motivated by regulations and consumer demands. Advanced High-Strength Steels (AHSS) offer the automotive industry the options to achieve both goals [1, 2].

AHSS combine high strength with good formability. This is achieved by balanced chemical composition and thermo-mechanical treatment during production of these

steels. However, certain issues have been reported concerning the weldability of AHSS in production. Keys to weldability are the mechanical strength of the welds and the adjacent heat-affected zone (HAZ) and the failure mode under different loading conditions [3-5].

The strength of AHSS is critical for achieving structural rigidity. The failure mode of welds determines the absorption of energy in construction under dynamic loading, as experienced in a crash. A lot of research has been published on the subject of improved weldability of AHSS [5-10].

Much less publicized is the time dependency of the mechanical properties of welds in certain classes of AHSS. Both strength and failure mode depend upon the time delay between production of the welds and mechanical testing of these welds. The most sensitive grades demonstrate strength variations of up to 100 % that take place within 24 h of welding. Such a time-dependent weld performance has very significant implications for the classification of weld performance.

This paper summarizes the initial findings of work carried out by Corus and Sumitomo Metals Industry (SMI), studying the time-dependent weld properties of certain AHSS. For the purposes of this paper, time-dependent changes in weld properties are termed "Ageing."

2 BACKGROUND

2.1 Strength and failure modes of welds

The increased strength levels of Advanced High-Strength Steels (AHSS) are the result of careful alloying and thermo-mechanical heat treatment during steelmaking. Both aspects are affected by welding. The carefully-designed microstructure is destroyed during welding. In the fusion zone, the melting and subsequent solidification of the material will wipe out all traces of the thermo-mechanical history of the material prior to welding. In the heat-affected zone (HAZ), the material characteristics change due to phase transformations, re-crystallization, grain growth, tempering, precipitation, etc.. Which effects will be produced and to what extent they will affect the material are dependent upon the thermo-mechanical history of the material prior to welding (e.g. cold-rolled vs. annealed material) and the thermal load of the material during welding [11].

As the level of alloying elements added to a steel is increased, the hardenability of the material increases accordingly. This hardenability is often expressed as a Carbon Equivalence (CE) number. There are various CE numbers published for different steels, accounting for different ranges of chemical composition of welds and cooling rates (e.g. different welding processes) [12-13]. Most famous of all CE numbers is probably the Carbon Equivalence number commonly referred to as the IIW CE number.

$$CE(IIW) = C + Mn/6 + (Cr + Mo + V)/5 + (Ni+Cu)/15 \quad (1)$$

This Carbon Equivalence number was published in 1967 [14]. The IIW CE number is usually used for steels with carbon levels exceeding 0,18 wt% C, as it was found that other carbon equivalent numbers worked better for steels with lower carbon content. In 1968, Ito & Bessyo published a paper in which a more complete relation was derived to predict post-weld hardness of steels containing Carbon levels of less or equal to 0,12 wt% [15]. The chemical portion of this formula (Pcm) is commonly used as a Carbon Equivalence number for steels with [C] < 0,18 wt%.

$$CE(Pcm) = C + Si/30 + (Mn + Cu + Cr)/20 + Ni/60 + Mo/15 + V/10 + 5B \quad (2)$$

The hardenability is increased in two ways: the minimum cooling rate to form harder microstructural phases (especially martensite and sometimes bainite) is decreased and the hardness of the resultant phases after cooling is increased.

Based upon work done by Blondeau et al. [16 & 17], Chaillet et al. [18] obtained equations that link the critical cooling rate for the formation of phases after welding to the chemical composition of high-strength steels:

Martensite:

$$\log V_{cr} = 7,42 - 3,13C - 0,71Mn - 0,37Ni - 0,34Cr - 0,45Mo \quad (3)$$

Bainite:

$$\log V_{cr} = 6,33 - 2,31C - 0,73Mn - 0,53Ni - 0,41Cr - 1,37Mo \quad (4)$$

where

V_{cr} is the critical cooling rate at 700 °C in °C/hour.

The element content is expressed in wt%.

Also equations were derived that link the post-weld hardness of the resultant phases after welding to the chemical composition of high-strength steels:

Martensite:

$$Hv = 97 + 949C + 27Si + 11Mn + 8Ni + 16Cr + 20\log V_{cr} \quad (5)$$

Bainite:

$$Hv = -348 + 185C + 330Si + 153Mn + 66Ni + 144Cr + 191Mo + \log V_{cr}(89 + 54C - 55Si - 22Mn - 10Ni - 20Cr - 33Mo) \quad (6)$$

The martensitic transformation is not time-dependent. Instead, it is temperature-dependent. At a certain temperature, M_s (martensite start temperature), austenite will transform into martensite. The driving force for nucleation of martensite at M_s is given by [19]:

$$\Delta G^{\gamma \to \alpha} = \Delta H^{\gamma \to \alpha} (T_0 - M_s)/T_0 \quad (7)$$

where

T_0 is the temperature where the free energy of the α' phase starts to exceed the free energy of the γ phase.

The temperature at which martensite starts to form is dependent upon the chemical composition of the material. Andrews [20] drew up an empirical equation

relating the temperature at which martensite starts to form, M_s, to the chemical composition of the material.

$$M_s = 539 - 423C - 30,4Mn - 17,7Ni - 12,1Cr - 11Si - 7Mo \qquad (8)$$

Martensite will form directly, as it is a military transformation, without delay. Thus, at a fixed temperature, fixed amounts of martensite have been formed. This will continue until another transition temperature is reached, M_f (martensite finish temperature), and martensitic transformation stops altogether. M_f may not correspond to 100 % martensite, as some retained austenite can be left below M_f. The retention of austenite may be due to high elastic stresses between the last martensite plates that are formed.

The formed phase proportion, p, during martensite transformations can be described by the Koistinen-Marburger model [21]:

$$p(\theta) = P[1 - \exp(b - (\theta - M_s))] \qquad (9)$$

For $\theta \leq M_s$ P represents the proportion obtained at infinitely low temperatures (usually associated with 1), and b characterizes the transformation rate.

Steven et al. [22] related fraction of martensite formed directly to the temperature below M_s, with M_f being 215 °C below M_s (see Figure 1).

It is not the hardness of the post-weld microstructure that determines the performance of the weld, but there appears to be a relation between the hardness of a weld and its mechanical characteristics. Chaillet et al. [18] empirically relate the hardness to the tensile strength:

$$F = 1,95 \times 10^{-4} \, HV^2 + 0,193 \, HV + 18,5 \qquad (10)$$

with [F] = kgf/mm^2 and HV the measured Vickers hardness.

Gould et al. [23] state that martensite, particularly with increasing carbon content, results in weld zones with hardness levels certain to fail in a brittle manner. In another report [24], such a failure mode is attributed to solidification-related porosity, which can contribute to the formation of critical-sized flaws that can eventually propagate down the faying surface. Harder microstructures then allow easier propagation of these flaws into cracks.

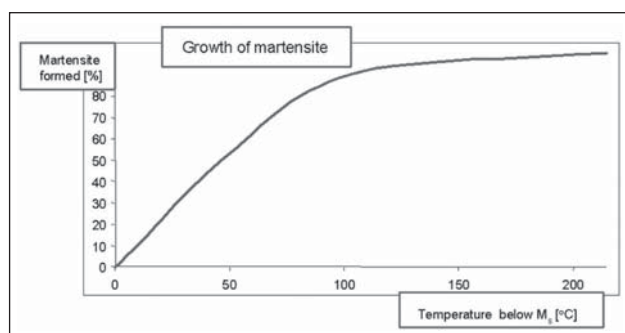Attempts have been made to relate weld failure mode directly to post-weld hardness and even chemical composition. Many studies on high-strength steels in the 1980s attempted to derive a modified carbon equivalent formula to define weldability, in particular, the borderline of potential interface failures for resistance spot welds. While reasonable correlation was achieved, no universal relationship was found. In addition, there is the question of whether a maximum weld hardness value could be specified to define the limit of suitable weldability. Although hardness levels around 400 HV and above are certainly more likely to give interface failure, there appears to be no definitive answer, as material thickness and material type can also have an effect [25].

Mimer et al. state that usually, welds with hardness exceeding 400-450 HV caused unstable fractures, and interfacial failures were seen in spots with weld hardness exceeding 450 HV [26]. Radaj [27] even mentions a common desire to aim for degrees of hardness below 350 HV in general in welding. He states that the problem is aggravated by diffusible hydrogen in martensite-hardened zones, leading to brittle fracture.

Internal research at Corus and SMI indicates a relationship between hardness and failure mode in resistance spot-welded joints. It is found that peel type loading of resistance spot-welded joints (e.g. coach peel, cross tension tensile and chisel testing) begins to produce partial plug and interfacial failures at hardness levels exceeding 450 HV, corresponding with the published reports mentioned above.

The approximate relationship between post-weld hardness and resistance spot weld failure mode in peel-type loading, as found by Corus and SMI, is illustrated in Figure 2. It can be seen that there are no set levels of hardness, where one type of failure mode changes to another type of failure mode. Instead, there is much overlap between the hardness levels, where specific failure mode types occur, again corresponding with the published reports mentioned above. This indicates that post-weld hardness is not the only factor determining failure mode. Indeed it is likely that other chemical and mechanical factors play important roles.

2.2 Ageing

"Ageing is a change in a metal by which its structure recovers from an unstable condition produced by quenching (quench ageing) or by cold working (strain ageing)" [28]. Or, *"a change in the properties of certain metals and alloys that occurs at ambient or moderate*

Figure 1 – Growth of martensite with cooling below M_s [22]
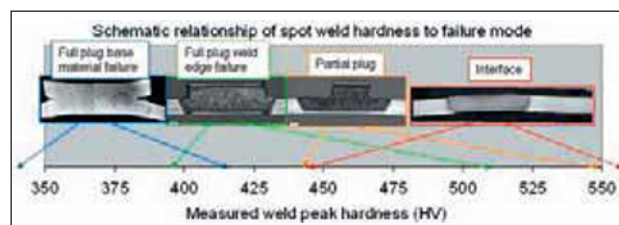

Figure 2 – Schematic relationship between resistance spot weld hardness and failure mode in peel-type loading

elevated temperatures after hot working of a heat treatment, or after a cold working operation. The change in properties is often, but not always, due to phase change (precipitation), but never involves a change in chemical composition of the metal or alloy" [29]. In this report, "ageing" is used to describe the change in mechanical performance of resistance spot welds over time.

The ageing effect in the resistance spot-welded materials described in this report was first noted in 2004, within the framework of the cooperative research project between Corus and SMI. After resistance spot welding, samples showed partial plug failure at low tensile strength when tested immediately after welding. However, a second batch of test specimens tested the next day showed full plug failure and much-increased strength levels. The weld hardness measured around 505 HV and therefore the joints were expected to show partial plug or interfacial weld failure. Instead, full plug failure was observed after delayed testing. Further investigation confirmed a time-dependent behaviour of the mechanical properties of the welds, Figure 3.

The ageing effect has been observed before. Sawhill *et al*. reported an "ageing"-like effect in 1979 [30]. Their observations were made after peel testing plain carbon and re-phosphorized steels. It was noted that welds made in oiled material and tested immediately after welding showed partial plug failure, with a small interfacial zone surrounding the weld nugget. Welds tested one hour after welding showed full plug failure. Welds made in material where the oil was removed prior to welding also showed full plug failure. It was concluded that weld fracture was probably assisted by hydrogen present in the weld and that the effect was reversible. However, this effect was not elaborated upon.

Mohrbacher [31, 32] discussed the causes and mechanisms of hydrogen cracking in steels with post-weld hardness exceeding 350 HV. Hydrogen absorption is caused by corrosion of steel in a promoter containing solution. Various promoters of hydrogen pick-up during welding were identified:

– anti-corrosive oils, lubricants and coolants;
– pollution and residues of packing materials;
– moisture;
– welding atmosphere and shielding gasses;
– weld filler material.

The latter promoters are not of importance in resistance spot welding, which was not the specific subject of the presentation. H and H_2 are said to re-combine at internal surfaces between the matrix and inclusions. This leads to a build-up of gas pressure in the defective area. Subsequent generation of tensile and shear stresses in the surrounding steel matrix may then lead to crack initiation. It was shown that the Erichsen index number for laser-welded TRIP steels increased over a period of 72 h. This improvement was attributed to the "effusion of hydrogen."

Improvement of mechanical characteristics in steels after hardening has been known for a long time. Bain [33] explained an increase of hardness in quenched steels due to transformation of austenite at low temperatures. *"The product immediately after the quench is by no means necessarily at a standstill."* He also proposed a slight hardening effect of an "ageing period" upon freshly-quenched steel, due to a slight reduction of stress as a result of slight movements within the metal, relieving stress and thus raising the yield point.

The occurrence of austenite after quenching in TRIP steels is likely because these steels have been alloyed to contain (retained) austenite at room temperature. There are several elements that influence the occurrence of retained austenite at room temperature. The $M_s – M_f$ range can be lowered. Pickering [34] reports values of a change of 450 °C per wt% carbon or nitrogen in Ms. Other elements are reported to lower M_s, though not by the same amount (see Table 1). Some of these elements are added in much larger quantities than carbon and can thus still play a very important role in the stabilization of austenite.

Local differences in chemical composition in the welds allow for the occurrence of retained austenite after quenching. Another cause for the occurrence of retained austenite is local stress concentrations which do not allow for the formation of martensite. The transformation from austenite to martensite is associated with ~4 % expansion. This causes stresses which suppress further growth of martensite in an austenite grain [19]. If possible, retained austenite decomposes at lower temperatures in fine carbides [35], or (at slightly elevated temperatures) into cementite and ferrite [36]. Decomposition rates are dependent upon chemical composition (there is diffusion of elements at room temperature), temperature and stress levels (relaxation).
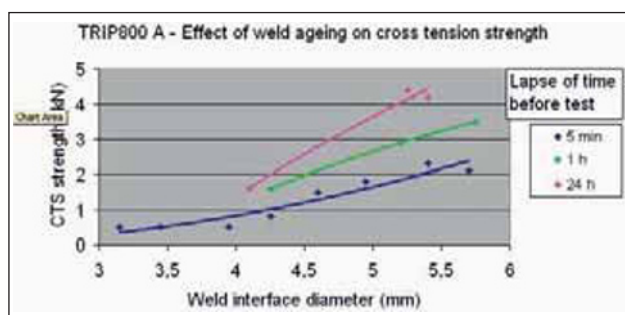

Figure 3 – The effect of ageing on cross tension strength in TRIP800 A

Table 1 – Change of M_s in °C per wt% of various elements

C	N	Al	Si	V	Mo	W	Cu	Mn	Cr	Ni	Co
-450	-450	-53	-50	-46	-45	-36	-35	-30	-20	-20	+10

3 OUTLINE

To investigate the ageing effect, a joint research project was set-up by Corus and SMI. The initial goal of the study was to determine exactly what the ageing effect is and how the weld properties change over time. This was done in a series of steps investigating various aspects of the ageing response. The results and discussion of the results are reported in this paper:

– Mapping the ageing effect

  • What exactly is the ageing effect and how do weld characteristics change over time?

– Process parameters

  • What are the effects of various process settings on weld ageing?

– Materials

  • What is the ageing response of different material types?

– Mechanisms

  • What is the effect of oil on ageing of spot welds?

  • Does hydrogen affect the ageing response?

  • Do phase changes affect the ageing response?

The report concludes with a hypothesis to explain the weld ageing mechanism in advanced high-strength steels. In addition, the implications of the weld ageing effect for standardized testing of weldability in advanced high-strength steels are discussed.

4 MATERIALS

Several materials were used to investigate the ageing phenomena (see Table 2). The materials were chosen to represent a cross-section of AHSS currently used for automotive applications. The materials differ in type, chemical composition and strength levels (600 MPa and 800 MPa).

The critical cooling rates at 700 °C to form martensite and bainite have been calculated for these steels according to Equations (3) and (4). The results can be seen in Table 3. The cooling rate at 700 °C in resistance spot welding has been measured and calculated for similar material thickness [12-13]. It was found to be far higher than the calculated values for the critical cooling rate for martensite. It can therefore be assumed that

Table 3 – Critical cooling rates at 700 °C

	t [mm]	$V_{cr, martensite}$ [°C/s]	$V_{cr, bainite}$ [°C/s]
DP600	1,5	229	21
DP800	1,2	89	8
TRIP800 A	1,2	166	18
TRIP800 B	1,2	185	19
TRIP800 C	1,2	133	21

the welds formed in the experiments are predominantly martensitic welds.

5 EXPERIMENTAL PROCEDURES

Resistance spot welding tests were performed on the 5 AHSS materials using the parameter settings shown in Table 4 below. The spot welds were opened by use of cross tension (CTS) or coach peel (LTS) testing, using sample dimensions shown in Figure 4.

The weld failure loads were recorded and the weld diameters were measured according to ISO 14329 [37]. In all cases in this investigation, where reference is made to spot weld nugget, plug, interface or partial plug failure modes, the ISO 14329 definition is used. When a fixed weld diameter is referred to, this implies the nugget diameter or the area of the fused zone. The nugget diameter is not affected by factors such as testing type, failure mode or cooling rate.

Many of the tests in this investigation required welds to be produced at a constant nugget diameter, so as to prevent harmful scatter which could influence the
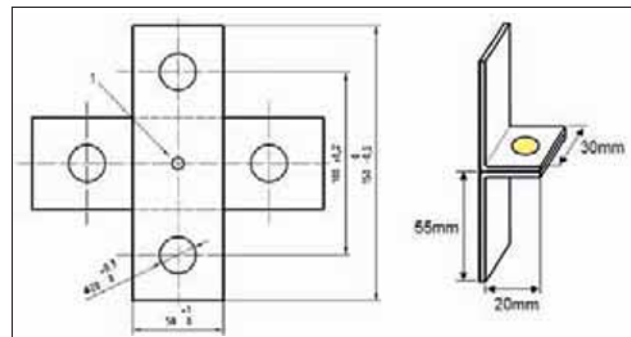
Figure 4 – Cross tension specimen (ISO 14272:2000 [38]) and coach peel test sample (LTS)

Table 2 – Materials used in weld ageing experiments

	t [mm]	Coating	Oil	C [wt%]	Mn [wt%]	Si [wt%]	P [wt%]
DP600	1,5	GI	Fuchs RP4107S	0,095	1,7	0,35	0,017
DP800	1,2	GI	Quaker N6130	0,135	2,1	0,3	0,014
TRIP800 A	1,2	-	S550	0,185	1,5	1,8	0,008
TRIP800 B	1,2	-	S550	0,17	1,5	1,7	0,01
TRIP800 C	1,2	GI	Quaker N6130	0,19	1,6	0,3	0,09

Table 4 – Welding parameter settings used

	DP600	DP800	TRIP800 A	TRIP800 B	TRIP800 C
Machine	MFDC 1 000 Hz				
Electrode	ISO 5821 'B' A2/2 16/6 mm				
Electrode force	4,5 kN				
Water cooling	4 l/min				
Weld time	400 ms	400 ms	400 ms	400 ms	400 ms
Weld current	7,3 kA	varied	6,0 kA	varied	7,1 kA
Weld nugget Ø	5,1 mm	varied	5,8 mm	varied	5,8 mm
Hold time	150/1 000 ms	1 000 ms	150/1 000 ms	1 000 ms	150 ms

results. In all cases, extreme care was taken to prevent any external factors from interfering with the welding process. All test welds were produced simultaneously in one production run, without interruption. The forces applied, welding currents and water cooling flow rates were monitored throughout testing. After destructive testing of the welds, the nugget diameters were measured to ensure that a constant weld size was used throughout the test series.

6 EXPERIMENTAL WORK

6.1 Mapping the ageing effect

Initial results had shown a very real effect of ageing upon the mechanical properties of welds, but the tests were not detailed. It was decided to clearly and carefully map the full ageing response, by testing many samples at close intervals over an extended time period. To achieve this, welds were made at constant parameter settings to obtain equal weld nugget diameters and to prevent this factor from influencing the results. Welding was carried out on material TRIP800 A (in as-received condition, 1 g/m^2 S550 oil) and the welding parameters used are given in Table 4. A hold time of 1 000 ms was chosen for this research, longer than that typically used by most OEMs.

After welding, cross tension testing was carried out at time intervals ranging from 30 s up to 28 h (testing was ceased at 28 h, as previous experience showed that no further measurable changes occurred after this time). Figure 5 shows the measured ageing response.

Despite some small amount of scatter in the results, trends were observed. Initially, weld strength was low, increasing to a peak at around 3–5 min (~180–300 sec), then dropping away to a dip at around 1 h (~3 600 s). The weld strength then increased again up to a final level at around 24 h (~90 000 s).

When the weld failure mode is examined, similar trends are observed (Figure 6). Under the welding conditions used in this test, ageing did not lead to full plug failures, but it did have an effect upon the size of the partial plug which was pulled in cross tension testing. After 30 s, large partial plugs were pulled which rapidly deteriorated to small partial plugs by 1 min, but increased to larger partial plugs again by 5 min. After 1 h, the partial plug size had again become smaller, at around 3–4 h the partial plug size increased, only to decrease again by 24 h. There are close links between the influence of ageing upon weld cross tension strength and failure mode, as the initial peaks and dips correspond to the same lapse of time. However, the final weld partial plug size did not correspond to the strength levels reached after 24 h.

6.2 Effect of process parameters on weld ageing

Spot weld ageing was known to occur over a range of weld nugget sizes, as can be seen in Figure 3. In this investigation, the effect of the cooling rate on the ageing response was examined.

Welding was carried out on material TRIP800 A (in as-received condition, 1 g/m^2 S550 oil) and the welding parameters used are given in Table 4. The parameters were fixed to prevent any variation in weld nugget diameter. The weld hold time was reduced to 150 ms

Figure 5 – Ageing response of 5,8 mm nugget diameter spot welds in TRIP800 A
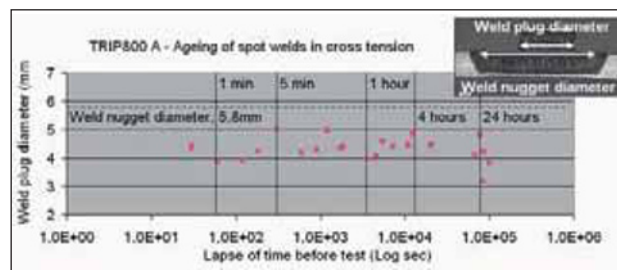
Figure 6 – Ageing response of 5,8 mm nugget diameter spot welds in TRIP800 A

which is more typical of automotive spot welding standards.

Just as before, the welds were opened by cross tension testing at time intervals up to 24 h after welding. The resulting cross tension strengths are compared to the 1 000 ms hold time results in Figure 7. The shape of the ageing response for the 150 ms hold test is similar to the result for 1 000 ms, although higher weld strengths are obtained. Both tests reach an initial peak weld strength after 5 min. However, the shorter hold time leads to a dip in weld strength after only 15 min, significantly sooner than when a 1 000 ms-hold is applied. After one hour, the weld strength has increased again and it continues to increase up to 24 h after welding. This explains why in initial testing (hold time 150 ms), higher weld strengths were measured after one hour (Figure 3), while when a 1 000 ms-hold was used, the weld strength dropped after one hour, as hold time influenced the time until the strength dip occurred.

The level of weld strength increase is roughly the same for both hold times: a 150 ms-hold increases from 3,8 kN at 30 s, up to 4,75 kN after 24 h, and, a 1 000 ms-hold increases from 3,4 kN after 30 s, up to 4,2 kN after 25 h. But the weld strength in the "dip" is lower for a 1 000 ms-hold, 2,7 kN, whereas with a 150 ms-hold, the weld strength only dips down to 3,8 kN. Once again, the pattern of weld failure mode (measured partial plug diameter) followed the same time-dependent pattern as the cross tensile strength.

When the hardness of the welds produced using differing hold times were compared (Figure 8), very little difference is seen. Indeed, the average hardness over the weld zone is 518 HV for a 150 ms-hold and 524 HV for a 1 000 ms-hold, a not so marked difference to separate from inherent measurement error.
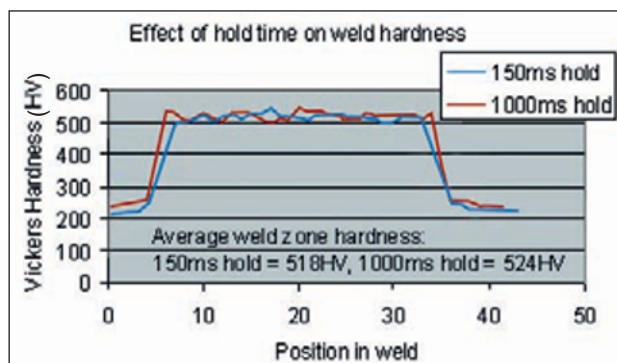
It appears that although hold time has an influence on the ageing response, this cannot be satisfactorily explained by a simple increase in weld hardness.

6.3 Ageing response of different materials

A DP600 steel was tested for ageing response with hold times of 150 ms and 1 000 ms. The welding parameter settings used can be found in Table 4, with the
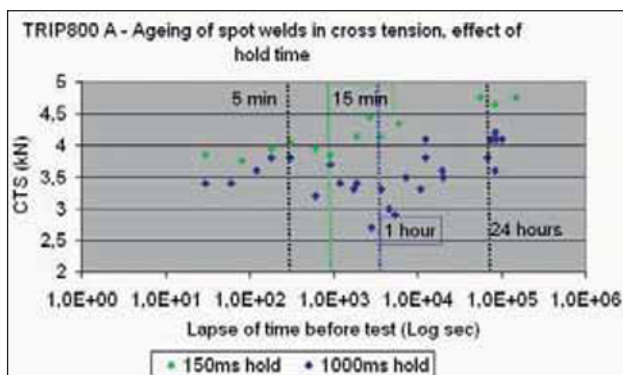

Figure 8 – Effect of hold time on weld zone hardness for 5,8 mm nugget diameter welds in TRIP800 A (samples taken 48 h after welding and mounted in epoxy at room temperature)

parameter settings constant for all welds so as to prevent any variation in weld nugget diameter influencing the test result. The DP600 material was welded in as-received condition with 1 g/m^2 Fuchs RP4107S oil.

Figure 9 shows how the cross tension strength of the welded joints varied with time. When a hold time of 150 ms was used, the measured weld strength did not show any significant evidence of ageing. However, in the case of the welds made with a 1 000 ms-hold time, an ageing response was indeed found. An initial strength peak occurred between 1 and 5 min and a strength "dip" occurred between 1 and 3 h after welding, with a final strength increase at ~24 h.

TRIP800 B and DP800GI were also tested for an ageing response. Both were welded in as-received condition (oiled) with the parameters given in Table 4 and the weld nugget diameter was varied. In Figure 10, it can be seen that both materials showed ageing-related strengthening in the welds. In previous testing, TRIP800 B had also demonstrated ageing when welded with conventional hold times (~100 ms).

The spot weld ageing response of TRIP800 C steel was also tested. The material was welded in as-received condition using the parameters given in Table 4, with a hold time of 150 ms. Unlike the results for TRIP800 A which showed ageing at both a 150 ms and 1 000 ms-hold, the TRIP800C material does not show any evidence of spot weld ageing, as shown in Figure 11. All welds in TRIP800 C failed as interface or partial plug.

Figure 7 – Effect of hold time on ageing response of 5,8 mm nugget diameter spot welds in TRIP800 A
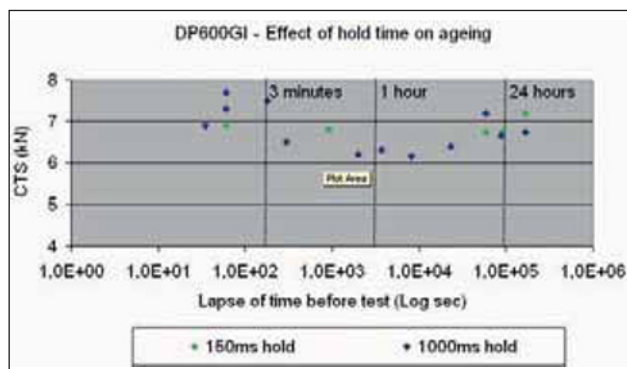
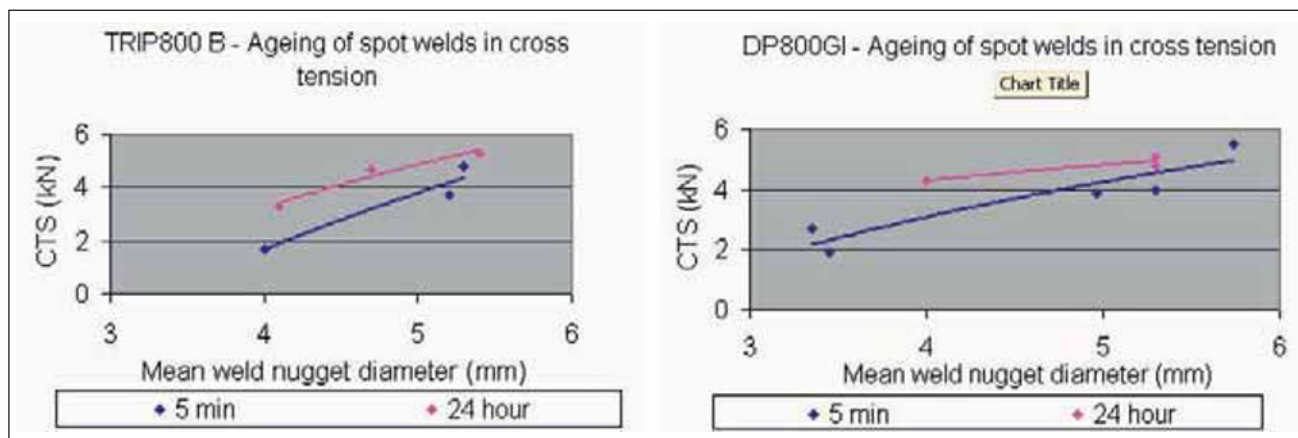**Figure 9 – Effect of hold time on ageing in 5,1 mm nugget diameter DP600GI spot welds**

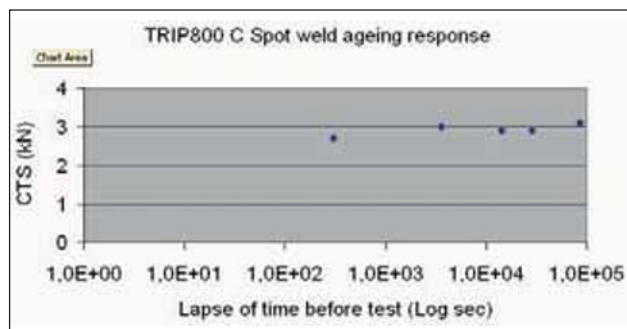**Figure 10 – Ageing effect in TRIP800 B and DP800GI**

Figure 11 – Ageing effect in 5,8 mm nugget diameter TRIP800 C spot welds

6.4 Effect of ageing on weld microstructure

Under normal welding conditions (a hold of 150 ms), spot welds in TRIP800 A gave partial plug failure modes in cross tension testing immediately after welding and, 24 h later, failed as full plugs. The failed samples were examined by optical microscopy, to study the fracture path through the weld microstructure. Immediately afterwards, welding failure occurred along grain boundaries, or more specifically, along the boundaries of the first dendrites, to form in the solidification structure. Twenty-four hours after welding, the same welds no longer broke on the dendrite boundaries, but outside the weld in the HAZ (heat-affected zone), as seen in Figure 12.

An EPMA analysis was performed to find the elements located on the dendrite boundaries. Figure 13 shows the EPMA maps produced in TRIP800 A. The dendrite
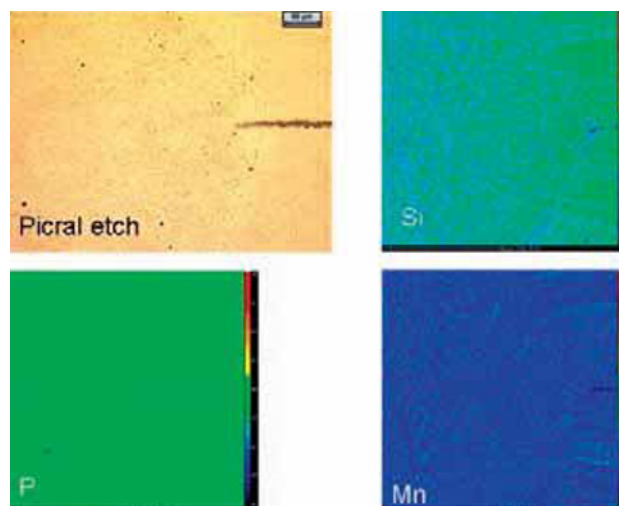
(Colour intensity shows elemental peak count, each map has a different intensity level.)

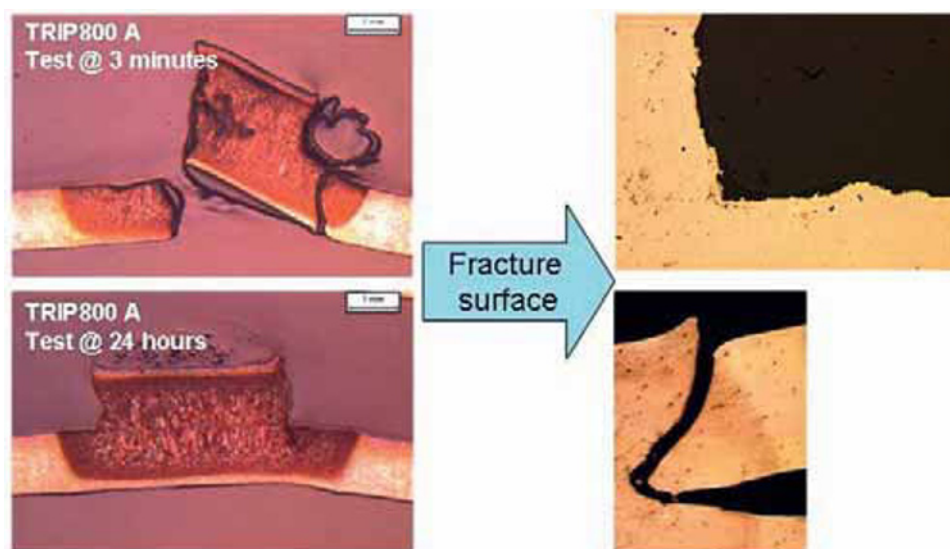**Figure 13 – EPMA analysis of TRIP800 A welds**

Figure 12 – Fracture surface of aged spot welds in TRIP800 A

boundaries can be clearly seen by heavy segregation of manganese and silicon. No significant phosphor segregation was found, even when a higher resolution line-scan was performed. When an EPMA map was performed on TRIP800 C, a very definite phosphor segregation pattern on the dendrite boundaries was observed (Figure 14).

6.5 Effect of oil on ageing of spot welds

6.5.1 Welding experiments

The effect of oil on spot weld ageing was examined. TRIP800 A was prepared by degreasing in an ultrasound, agitated, acetone bath. After degreasing, the samples were immediately baked at 180 °C for 30 min, to further remove or evaporate any remaining oil or moisture that could be a possible source of hydrogen. Once the samples had cooled, welds were immediately produced to minimize any new accumulation of hydrogen from the atmosphere.

The weld samples were made with the same welding parameters as for the previous tests, with "as-received" (oiled) material, so that the results could be directly compared (see Table 4 for welding parameters).

Figure 15 shows how the weld cross tension strength of the degreased – baked samples compared with the "as-received" (oiled) samples over 24 h after welding. The first observation is that the degreased baked material produced stronger welds than the oiled material; the second, that the degreased baked material also showed an ageing response. The weld strengths measured were all higher than measured for the oiled material. The timing of the ageing response was also identical to the oiled material, with an initial strength peak at ~5 min, then a strength dip at ~1 h and a gradual strength increase up to 24 h. However, after 24 h, the degreased baked samples did not demonstrate higher strengths than were measured at the first peak (5 min), whereas the oiled samples showed higher strengths after 24 h, nearly obtaining the same final strength as the degreased baked samples.
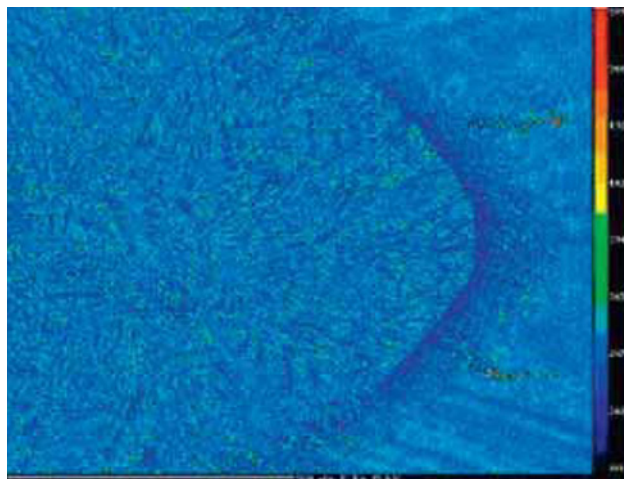
Samples of oiled and degreased TRIP800 A were examined using optical microscopy and hardness profiles were also produced, as shown in Figure 16. When the Picral-etched samples were examined, it was found that the degreased sample contained many finely-dispersed inclusions, located along dendrite boundaries. The as-received (oiled) sample contained fewer inclusions, although they were very large in size.

A second significant difference between the two samples was the weld zone hardness, the as-received (oiled) sample being harder than the degreased sample. Weld zone hardness could increase if components from the oil such as carbon dissolved into the weld [16-18].

6.5.2 Cathodic charging experiments

In order to test the theory that hydrogen can reduce the strength of welded joints, a simple experiment was performed. Spot welds were made in TRIP800 B and allowed to age and after 24 h the peel strength had increased. A second batch of welds was charged with hydrogen (Figure 17) and when these welds were tested, the peel strength was greatly reduced, with higher hydrogen levels giving the lowest weld strengths. Although this experiment does not prove that hydrogen is responsible for weld ageing, it demonstrates that higher hydrogen levels do reduce spot weld strength.

Oil on the sheet surface was tested as a possible source of hydrogen by thermal desorption analysis and the hydrogen gas which evolved was measured by mass spectrometry. Figure 18 shows the evolution of diffusible hydrogen for spot welds in material TRIP800 B, in "as-received" condition ($1 g/m^2$ S550 oil) and degreased (with acetone).

In the degreased weld sample, very little diffusible hydrogen was evolved, whereas the "as-received" weld sample contained a lot of diffusible hydrogen.

More spot weld samples were produced in "as-received" (oiled) condition; the diffusible hydrogen in the welds was measured. One sample was measured immediately after welding; this sample was quenched in liquid nitrogen 3 s after welding. The sample was
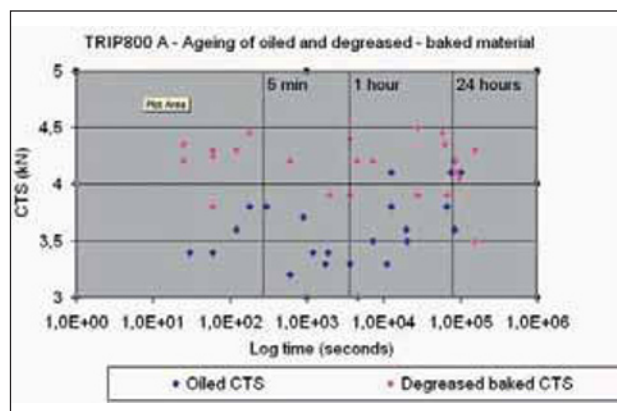

Figure 14 – EPMA phosphor map of TRIP800 C

Figure 15 – TRIP800 A, ageing of degreased – baked samples compared to as-received material (5,8 mm nugget diameter)
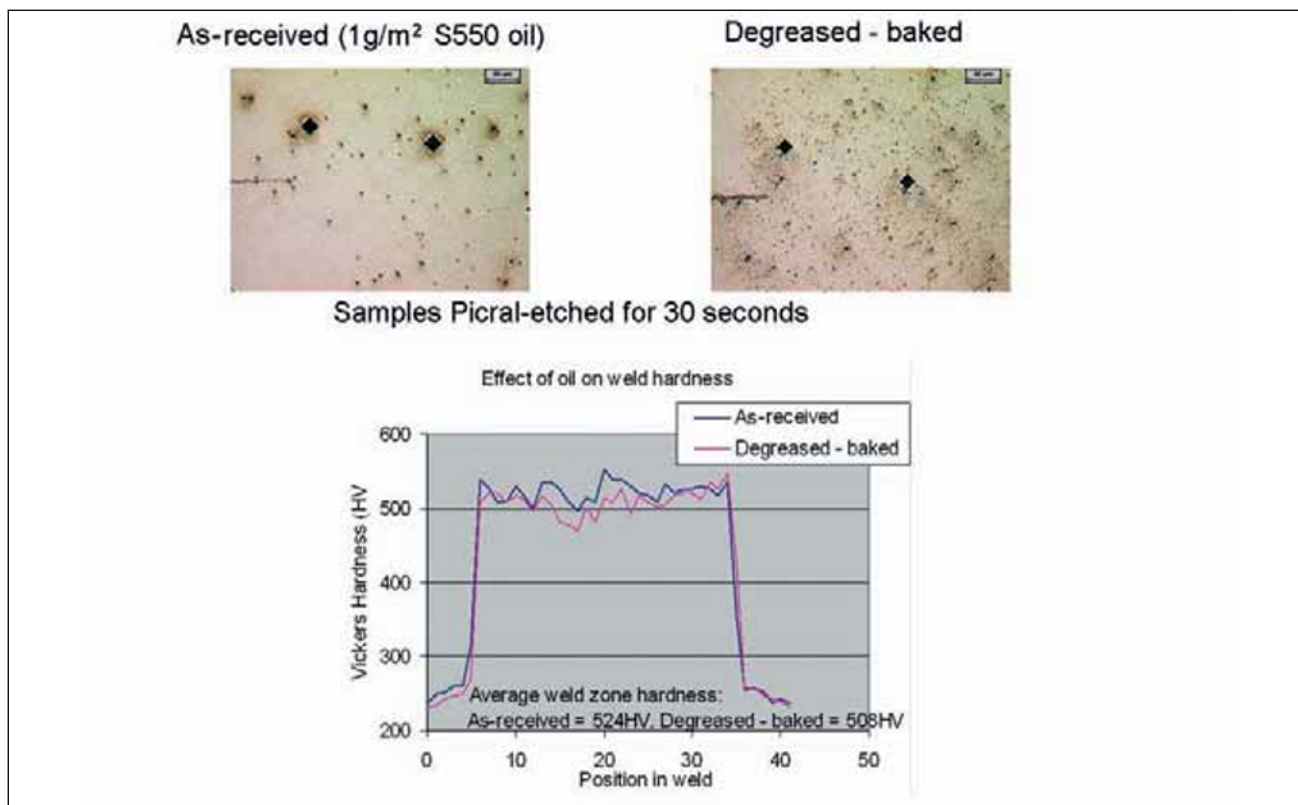
Figure 16 – 30-s Picral-etched welds in TRIP800 A and hardness profiles
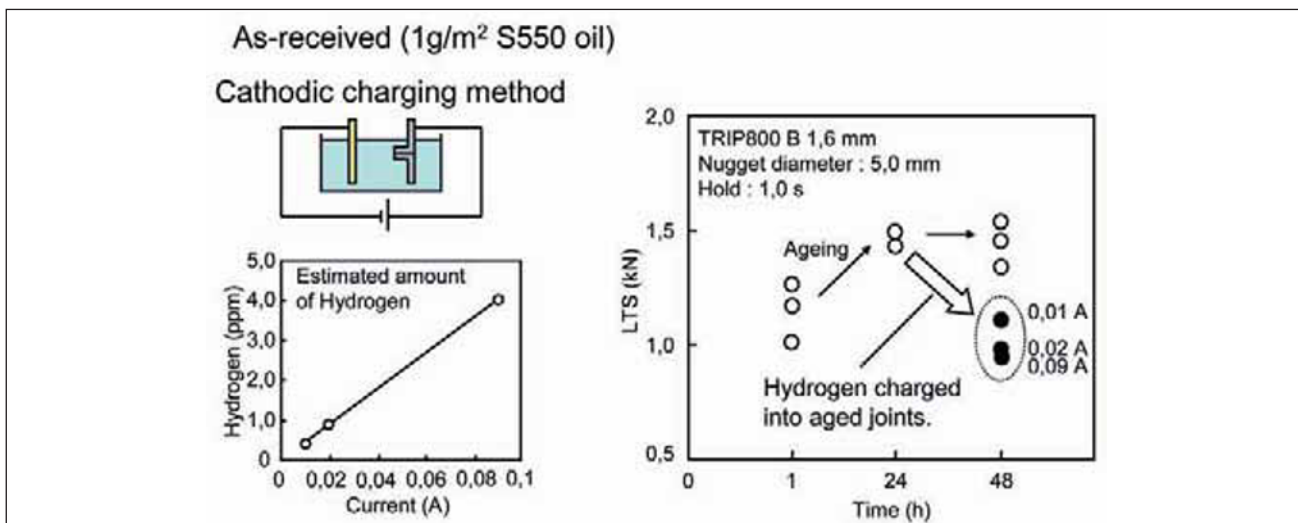

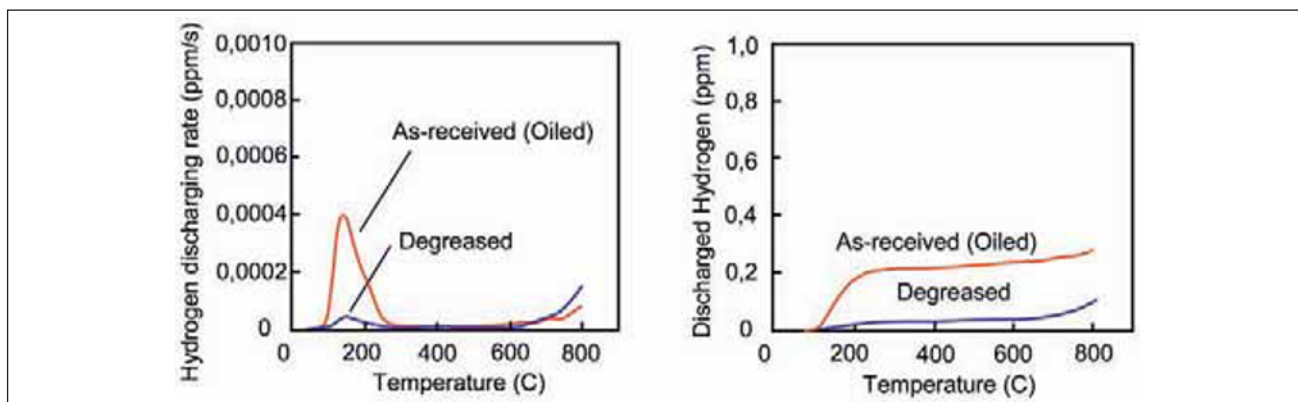**Figure 17 – Demonstration of how hydrogen charging can reduce weld strength in TRIP800 B (5,0 mm nugget diameter)**

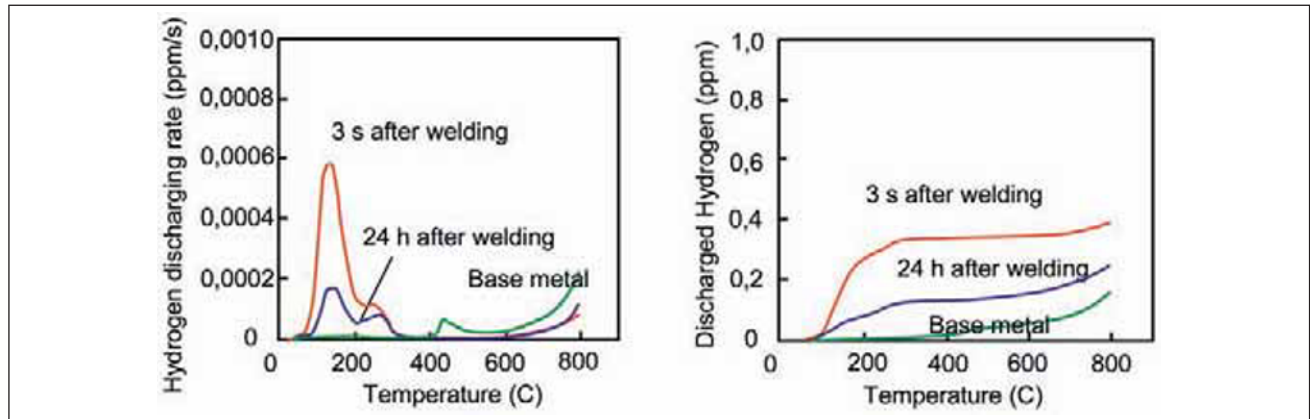**Figure 18 – Hydrogen measurements in TRIP800 B spot welds**

Figure 19 – Hydrogen evolved from spot welds in TRIP800 B

kept in liquid nitrogen during transport to the measuring equipment, the low temperature preventing hydrogen diffusion out of the weld. The hydrogen evolved was measured immediately after welding and 24 h later, as shown in Figure 19. It can be seen that immediately after welding, much hydrogen evolved and after 24 h, a decrease in hydrogen evolution was measured. These results indicate that hydrogen evolution from the weld zone is time-dependent and occurs on the same timescale as the ageing effect. The base material measurement demonstrates that the hydrogen does indeed come from the weld.

6.6 X-ray diffraction measurement of phases in weld zone

A weld sample was produced in TRIP800 A in "as-received" condition, a cross-section was prepared at room temperature and submitted for micro X-ray diffraction measurement with an area sensitive detector. A number of scans were made across the weld zone (resolution 800 μ), performed at 0, 2, 4, 6 and 24 h after welding.

The phases measured in the weld were identified on the 2 theta scale as predominantly Ferrite (actually martensite as the diffraction signal is the same), some Fe_3Si eutectic phase and austenite, as in Figure 20. The measurements taken between 0 and 24 h show no difference in the proportion of each phase measured.

A further measurement was taken 20 days later, shown in Figure 21. After 20 days, no austenite was found and in its place, tetragonal martensite had formed. This phase change is a further effect that could influence weld ageing.

Unfortunately, the X-ray measurements do not show if the transformation of austenite to martensite occurred immediately after welding, or very slowly over a long period of time. This is because the procedure for preparing the samples for X-ray analysis is likely to have influenced the results of the measurements. The weld samples were cut open with a water-cooled, rotating disk cutter (at a low rotation speed), then polished slowly by hand. The sample preparation was performed in such a way as not to bring too much heat or strain into the microstructure, since heat and strain could easily trigger phase transformations.

In reality, it was not possible to prepare the sample quickly enough in a manner that would not trigger some transformation, which means that the initial phase measurements were probably not consistent with what would normally occur in a weld.
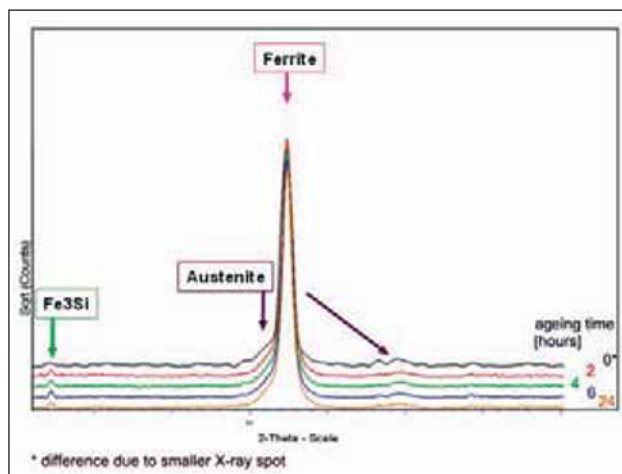

Figure 20 – Phases identified by X-ray diffraction in TRIP800 A
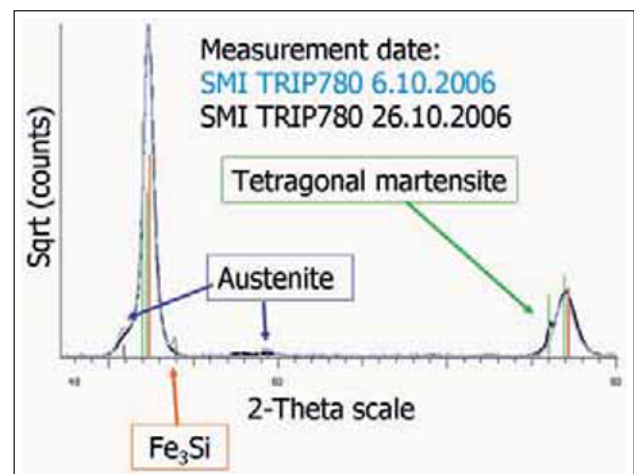

Figure 21 – Phases in TRIP800 A after welding and 20 days later

7 DISCUSSION

7.1 The ageing response

All care was taken to prevent scatter in weld nugget diameter from influencing the results, although some limited scatter is inherent in the resistance spot welding process. The time-related pattern of the ageing response showed the same characteristics in all tests where the effect was observed (Figures 5, 7, 9 and 15). The reproducibility of the ageing effect across a range of testing conditions and materials is a strong indicator that the variations in mechanical properties were influenced by more than an inherent variation in weld diameter.

When the ageing response of AHSS spot welds was studied in detail, it became clear that ageing was not simply an increase in weld strength with time. The following patterns were consistently observed:
– Immediately after welding, the cross tension strength was low.
– After 3–5 min, the cross tension strength had increased to an 'initial' peak.
– After the 'initial' peak, the strength had reduced, an event which was named a strength 'dip.'
– After 24 hours, the strength had increased to its maximum value.

The weld failure mode followed the same time-related pattern as weld cross tension strength, although some exceptions were observed. An ageing response was measured in spot welds in a variety of AHSS materials, DP600, DP800 and TRIP800.

7.2 The effect of hold time

Certain process parameters were able to influence the weld ageing response. Longer hold times reduced the cross tension strength achieved, regardless of ageing (Figure 7). Longer hold times were also able to promote ageing in a DP600 material, where no ageing occurred with conventional hold times (Figure 9). Hold time influenced the ageing response, but had no measurable effect on the weld zone hardness (Figure 8). Previous investigation had shown that increasing spot weld hold time beyond standard settings did not have an effect upon the spot weld cooling rate through solidification [5]. However, it is possible that longer hold times increase the amount of under-cooling and the level of internal stresses.

7.3 The effect of alloying elements on ageing

Weld ageing was only observed in AHSS, where spot weld microstructures are predominantly very hard martensite [22]. The link between alloying elements and martensite hardness was discussed by Blondeau *et al.* [16, 17]. In order for ageing to have a measurable effect on weld strength, a weld microstructure must be dominated by very hard martensite. The chemical composition is an important factor to determine the occurrence of hard martensite.

DP600 is less heavily-alloyed compared to DP800 and TRIP800, the latter having an alloy content designed to retain some austenite at room temperature. DP600 showed no weld ageing when conventional weld settings were used, suggesting that this material is not highly-alloyed enough to age. But when an extended hold time (1 000 ms) was used an ageing response was observed.

As with DP600, ageing was measured in DP800 when a 1 000 ms-hold time was used. The TRIP steels demonstrated an ageing effect when welded with conventional hold times (150 ms). The higher alloy content of the TRIP steels leads to the formation of a harder martensite phase in the weld and an increased chance of retained austenite at room temperature.

Optical microscopy of failed welds revealed that fracture occurred along dendrite boundaries in the weld zone before ageing and in the HAZ after ageing had taken place. Ageing is an effect that occurs on dendrite boundaries, which means it could potentially be influenced by segregation on those boundaries. No measurable ageing response was observed in TRIP800 C, despite a high level of alloying additions designed to retain austenite at room temperature. Spot welds in TRIP800 C steels always failed at low loads with partial plug failure modes. EPMA analysis of TRIP800 C welds showed heavy phosphor segregation on dendrite boundaries in the weld zone. Phosphor segregation reduces grain boundary fracture toughness and could negate any positive effect of ageing in high phosphor steels.

7.4 The influence of oil (hydrogen) on weld ageing

The cathodic charging experiments were able to show that increasing the hydrogen level in spot welds decreased the weld failure strength (Figure 17). The link between hydrogen and weld performance was demonstrated, with the highest hydrogen levels leading to the lowest weld strengths. It was also demonstrated that surface oil was the greatest source of diffusible hydrogen with the potential to dissolve in the weld zone (Figure 18). In a martensitic microstructure, the areas most susceptible to hydrogen-related cracking are prior austenite grain boundaries [36], which are precisely where failure occurs immediately after welding (Figure 12).

When welds were examined, it was shown that the highest level of diffusible hydrogen could be evolved immediately after welding; after 24 h, the level of evolved hydrogen had decreased (Figure 19). These experiments demonstrate that hydrogen is indeed picked up during welding and, over a 24 h period, diffuses out of the weld zone. The reduction in diffused hydrogen leads to increased weld strength. There is a strong relationship between hydrogen diffusion and weld ageing and a likely source of hydrogen is surface oil.

When the influence of oil was tested experimentally, it was observed that oiled welds had lower weld strengths than degreased welds (Figure 15). The lapse of time between the peaks and dips of the ageing response was not affected by the presence or absence of oil. After 24 h, the strength of the oiled welds had increased up to almost the same level as the degreased welds. This correlates very well with the hydrogen measurement results which show that hydrogen levels were much lower after 24 h, with lower hydrogen levels corresponding to higher weld strengths.

The fact that the lapse of time between the peaks and dips of the ageing response was not affected by the presence of oil in the welds (diffusible hydrogen) demonstrates that hydrogen is not the only mechanism of weld ageing. The ageing effect is probably the result of an interaction of two or more mechanisms.

Hydrogen is not the only component from oil that has a measurable effect upon the weld zone. Optical microscopy revealed that degreased welds had a fine dispersion of inclusions located on the dendrite boundaries. Oiled welds had fewer, but much larger inclusions [Figure 16]. Additionally, the hardness of oiled welds was higher than that of degreased welds.

The study of the effects of oil is ongoing, but it appears that the components of surface oils contribute more than just hydrogen to the weld zone; dissolved carbon would have a significant hardening effect. The increase in hardness resulting from oil dissolution in the weld is also likely to increase its susceptibility to ageing.

7.5 The role of phase changes in ageing

In X-ray diffraction measurements of welds in TRIP800 B, a small fraction of retained austenite was found immediately after welding (Figure 20). When the same welds were tested 20 days later, the retained austenite had disappeared, to be replaced by a tetragonal martensite phase (Figure 21). The results do not reveal when austenite transformed into martensite. It is known that over time, meta-stable austenite can transform to martensite at room-temperature. The mechanisms by which this can happen were already explained in 1939 by Bain in his famous lectures.

8 PROPOSED MECHANISMS OF AGEING

From the results of this investigation, it is concluded that there is a link between the presence of hydrogen in the weld zone and the cross tensile strength of a welded joint. It appears that the level of hydrogen in the weld decreases over time, causing an improvement in the cross tensile strength of the welded joint. Not only does the cross tensile strength of the joint improve, but the weld failure mode also improves. Just after welding, the welds containing hydrogen fail in a brittle manner, leaving a partial plug. Over time, the failure

mode improves and the plug size grows. Eventually, full plug failure can be achieved.

Possible sources of hydrogen have been identified in literature. Some likely candidates in resistance spot welding for automotive applications appear to be oils and lubricants, present on the materials as residue from previous production stages (protection against corrosion, packing and forming operations). The experiments reported in this paper clearly indicate a strong influence of oils on the surface to weld ageing. Of course, there are other possible sources of hydrogen (e.g. moisture on the surface).

It is uncertain in which way the presence of hydrogen affects the weld's capability to bear a load. It has been suggested that hydrogen concentrates around defects in the matrix of the welded joints (e.g. inclusions, grain boundaries). Even minor lattice defects (e.g. vacancies and dislocations) have been identified as possible hydrogen traps. There, the concentrations of hydrogen atoms collapse into micro-voids which can serve as both crack initiation points and propagators of cracks.

Another mechanism can be found in the solubility of hydrogen in the matrix itself. The solubility of hydrogen in the liquid phase is far higher than it is in solid steel [39]. After solidification, the decrease in solubility of hydrogen in austenite (TRIP) or δ-ferrite (DP) leads to super-saturation. On further cooling, the solubility of hydrogen in the matrix decreases (on transformation of δ-ferrite to austenite in DP steels, the solubility of hydrogen increases, but it decreases again with decreasing temperature). When austenite transforms into martensite, the solubility of hydrogen decreases even further. The mobility of hydrogen in the steel matrix at elevated temperatures is high and it can be assumed that much of the hydrogen can escape from the matrix. However, as the weld cools to room temperature, the mobility of hydrogen decreases.

After cooling, retained austenite will be super-saturated with hydrogen because of the hydrogen trapped at higher temperatures. This hydrogen cannot escape immediately at lower temperatures, due to decreased mobility and the fact that the retained austenite is surrounded by hydrogen-saturated martensite, blocking the escape routes for hydrogen. Park *et al*. identify these islands of retained austenite in a martensite matrix as bulk hydrogen traps [40]. Upon loading, plastic strains force the retained austenite to transform into martensite, releasing hydrogen to initiate cracks and cause undesired failure modes.

Although the mobility of hydrogen at room temperature is much lower than it is at elevated temperatures, over time, hydrogen would be able to leave the weld, thus reducing its detrimental effect upon the performance of the welds in loading.

The experiments indicate that hydrogen is not the only cause of the ageing effect. Another likely source is the decomposition of retained austenite. Retained austenite is always present after the formation of martensite, but in TRIP steels, the occurrence of retained austenite

after welding (and the amount of austenite present) is even more likely due to their chemical composition. The retained austenite forms pockets of decreased mechanical performance in the martensitic welds, leading to low weld strengths and undesirable failure modes.

The retained austenite is not stable. There is diffusion of elements, changing local chemical composition and there is relaxation of local stress levels which allows for the decomposition of austenite into bcc iron phases. Over time, the weld will thus gain in strength and lose certain possible paths for crack propagation.

9 THE IMPLICATIONS OF WELD AGEING FOR STANDARDIZED TESTING

Under normal practices, welds are tested mechanically soon after welding, whereas in reality, no automobile is placed on the open market until more than 24 h after welding.

Although not all AHSS materials exhibit the phenomena of weld ageing, the effect can be seen in a significant portion of AHSS grades.

When weld ageing does occur, the weld mechanical properties vary significantly over the first 24 h after the weld was made. This time-related behaviour has very serious implications for standardized testing procedures. A weld tested immediately after welding may produce low weld strengths and poor failure modes, leading to the conclusion that the material does not have an adequate weld performance. Whereas the same welds tested 24 h later would have been accepted.

The problem is that most welding standards do not specify a timescale between welding and mechanical testing. Many standards do not specify if a material should be welded in as-received (oiled) condition or degreased, but oil has a very significant effect on weld performance in ageing sensitive materials.

The weld schedule used is also of great importance. If very long hold times are applied, then ageing can be induced in materials where no ageing effect would normally be found (such as DP600). If the weld schedule used for material acceptance is not the same as the one applied in production, then further disparities could occur between measured results.

10 CONCLUSIONS

The experiments carried out demonstrate that the ageing effect is a very real phenomenon. However, ageing does not occur in most low carbon steels under normal welding conditions. With increasing levels of alloying additions, steels can become more sensitive to ageing and less rapid cooling rates are required to activate the effect.

In ageing sensitive materials, the spot weld mechanical properties can fluctuate significantly within 24 h of wel-

ding. In general, weld ageing leads to an improvement in mechanical properties, a factor that should be taken into account in standardized weld testing.

Although much is still unknown about the mechanisms of ageing and what really happens within the weld microstructure after welding, this investigation was able to reveal certain important characteristics of the ageing effect:
– Ageing is an effect that increases grain boundary fracture toughness in the weld zone.
– The ageing response leads to an initial peak in weld strength, followed by a dip and a final strength increase at around 24 h after welding.
– A measurable ageing effect occurs in certain highly-alloyed steels that combine a high weld zone hardness and low phosphor content.
– Oil on the sheet surface acts to increase the level of the weld ageing response.
– Oil has a negative influence on weld properties.
– Hydrogen from surface oil contributes to weld ageing.
– Other components of oils also affect the weld microstructure by increasing the weld hardness and influencing the size and distribution of inclusions.
– Based upon the results of this investigation, a hypothesis has been proposed to explain the ageing phenomenon.

ACKNOWLEDGEMENTS

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