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# Effect of partial martensite transformation on bainite reaction kinetics in different 1%C steels

SMANIO Véronique [a], SOURMAIL Thomas [b]

Ascometal-CREAS (research centre) Metallurgy, BP 70045 57301 Hagondange Cedex, France

[a]v.smanio@ascometal.lucchini.com, [b] corresp. auth: t.sourmail@ascometal.lucchini.com

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**Abstract.** Isothermal bainitizing of high carbon steel is used to obtain exceptionally high mechanical characteristics together with near zero distortion during heat-treating. Such heat-treatments are often carried out at temperatures close to but above the martensite start temperature, where transformation kinetics are unfortunately very sluggish. The formation of a small amount of martensite prior to isothermal holding is known to accelerate transformation kinetics. However, the influence of the amount transformed remains to clarify, and the consequence on final properties have not been explored.

In the following, the influence of partial martensite formation on the kinetics of bainite formation is investigated in 100Cr6 (AISI 52100) and similar grades. It is shown that, although the formation of martensite allows for shorter heat-treatment durations, it does not seem to affect the bainite kinetics, inasmuch as the bainite formation kinetics after formation of X% martensite are identical to that which would be expected if the first X% transformation had been obtained through isothermal bainitic transformation.

## Introduction

Isothermal treatment in the lower bainitic range is sometimes used in industry to obtain a combination of high fatigue strength and dimensional stability, or to reduce distortions associated with quenching of large, very high strength components. Steel grades typically concerned include 100Cr6 (AISI 52100), higher hardenability variants, and other high Si high C steels [1,2], although some low temperatures bainitizing trials have been reported on spring steels [3,4].

At low temperature, bainitic transformation can be very long resulting in high energy consumption and long production period. Different solutions have been investigated to shorten the transformation durations. A first category of solutions consists in modifications of the steel composition. In this regard, additions of Al and Co have been shown to be effective [1]. Unfortunately, Al additions are avoided in alloys requiring high cleanliness (as would typically be the case for the applications considered), and the amount of Co required (~2 wt%) renders the solution economically unviable. A second category of solutions consists in heat-treatment modifications. Various two-steps heat-treatments have been investigated by Vetters et al. [5,6] including partial pre-quenching followed by bainitizing. However, the latter was not investigated in details, presumably because initial results tended to show little effect on the transformation kinetics.

In the present work, the influence of martensite on bainite transformation kinetics is investigated using dilatometry. After identification of the martensite fraction as a function of temperature, different interrupted quenching treatments were carried out, followed by isothermal bainitizing at 220 and 250 °C.

## Experimental procedure

### Materials and specimens

The bearing steel 100Cr6 and a high silicon high carbon experimental grade were studied. Chemical compositions (Table 1) of all steels were analyzed by optical emission spectrometry (OES), except for carbon and sulfur (LECO analysis).

**Table 1: Chemical compositions of the steels (wt.%)**

| Steel grade | C | Si | Mn | S | P | Cr | Mo | Co |
|---|---|---|---|---|---|---|---|---|
| **100Cr6** | 1,01 | 0,24 | 0,30 | 0,003 | 0,012 | 1,50 | 0,05 | 0,00 |
| **100CoCrSi10** | 0,91 | 1,23 | 0,93 | 0,008 | 0,010 | 1,26 | 0,04 | 2,50 |

Industrially manufactured as-rolled bars (Ø75 mm) were used for the 100Cr6 grade. To obtain the high Si, high Co steel, a 40 kg ingot was manufactured using vacuum induction melting. The ingot was then hot forged to bars of 40 mm diameter. All dilatometric measurements were carried out on cylindrical samples of 12 mm length and 3 mm diameter machined taken at mid-radius of the bars.

### Heat treatment cycles

Dilatometric experiments were carried out on a Bähr 805 quenching dilatometer. During the dilatometric experiments the sample was inductively heated in vacuum and quenched or cooled using nitrogen. A schematic illustration of the heat treatment cycles used in the present study is given in Figure 1.

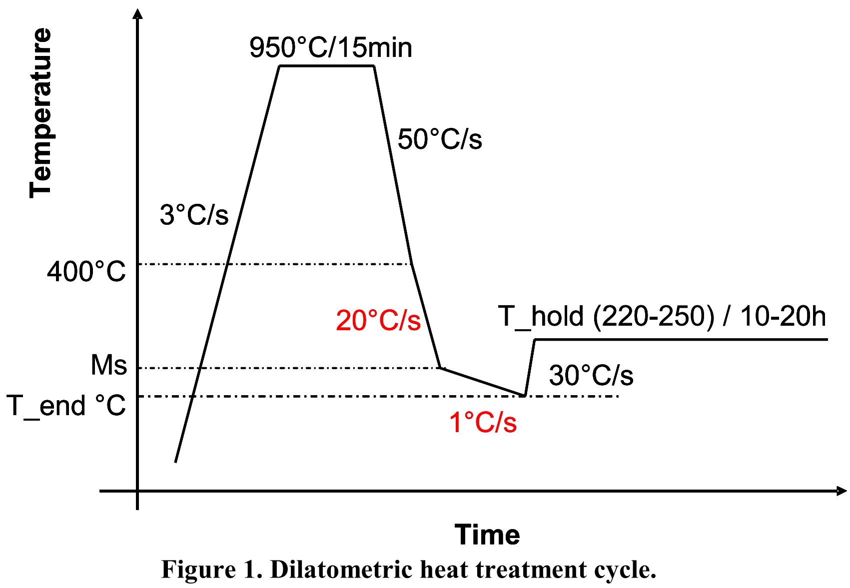


**Figure 1. Dilatometric heat treatment cycle.**

As illustrated, samples were cooled from the austenitisation temperature to a calculated end quench temperature (T_end), below the martensite start temperature (Ms). This allows formation of a given fraction of martensite before the isothermal holding step. Two different isothermal holding temperatures (220 and 250°C) were used; in each case, the heat-treatment was also carried out without martensite formation (standard bainitizing) so as to provide a reference. The cooling rate was decreased when approaching the end quench temperature to ensure the absence of thermal gradient within the sample.

In most of the experiments, the martensite finish temperature (Mf) was unknown because significantly below room temperature. Therefore, conversion from dilatometric measurements to martensite volume fraction was not as trivial as is the case when the transformation is complete at room temperature.

In order to estimate the volume fraction of martensite formed as a function of temperature, samples of both grades were quenched to room temperature. The recorded length change was then compared to calculations carried out assuming that martensite fraction followed the Koistinen-Marburger equation (Eq. 1, λ being considered a fitting parameter) [8], and using published data for lattice parameters of martensite and austenite [9].

$$Vf=1-\exp(-\lambda(Ms-T)) \quad (1)$$

The end quench temperature was thus estimated to provide 15 and 30% martensite in both steel grades. As shown in Figure 2, measured and calculated length changes were in good agreement except for the onset of transformation. The issue of volume fraction estimation will be discussed later.

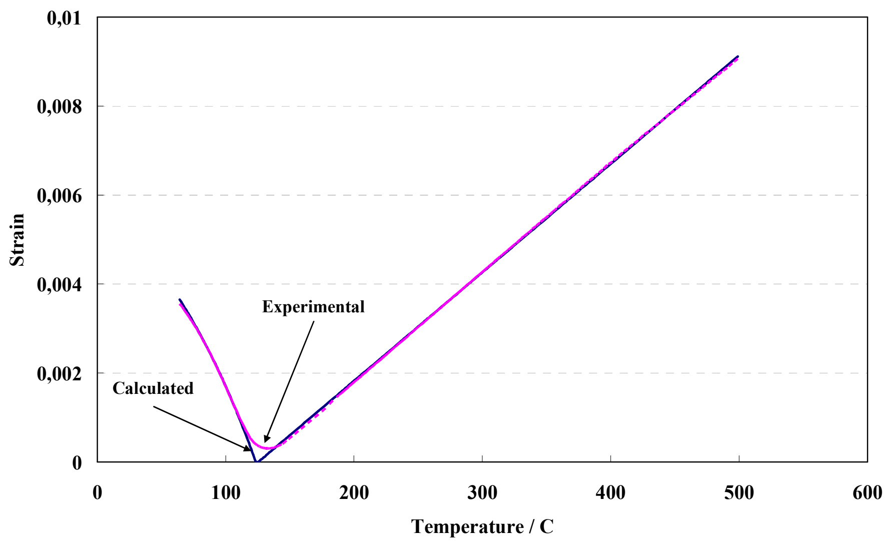


**Figure 2. Experimental and calculated length change versus temperature for 100Cr6.**

### Hardness measurement and retained austenite content

The hardness of each dilatometric sample was measured using a Vickers indenter and a load of 30 kg. Each value is given as the average of 3 measurements. Residual austenite content was determined using X-Ray diffraction in a Bruker D8 Advance diffractometer. A total of 5 peaks were scanned ({111}{γ}, {200}{γ}, {220}{γ}, {110}{α}, {200}{α}) using 12 different incident beam conditions (2Φ, 6Ψ) to minimize the influence of texture.

## Results

### Dilatometric experiments

Figure 3 shows the kinetics of the bainitic transformation for 100CoCrSi10 at 250 C with 0%, 15%, and 30% of martensite. To quantify the effect of martensite formation on the bainite transformation kinetics, the recorded length change for 15 and 30% prior martensite formation were shifted in time and strain, so as to minimize the difference with the reference signal (Figure 3).

The procedure was repeated for both grades, and for both heat-treatment temperatures (220 and 250 C). This is significantly more robust than comparisons based on transformation end time, as the latter is difficult to define when there is no horizontal tangent in the signal (as is the case at 250 C).

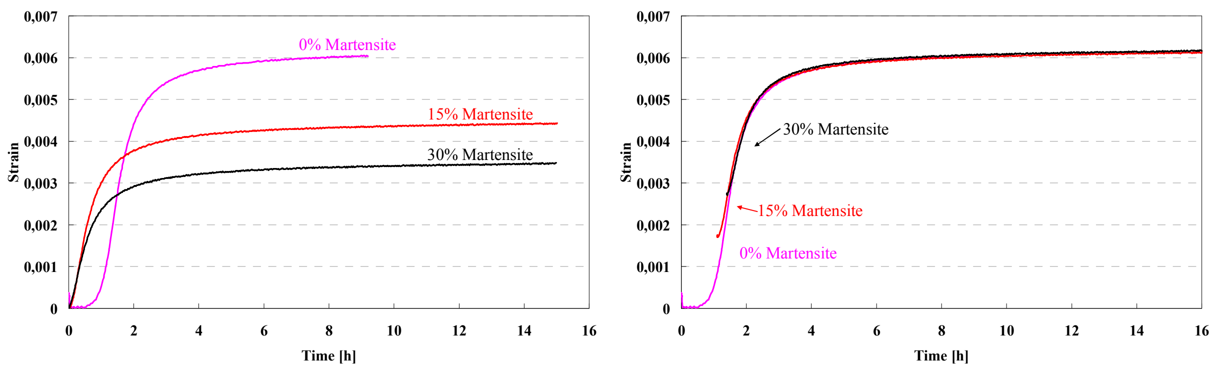


**Figure 3. Bainitic transformation of 100CoCrSi10 at 250°C, as measured and after shifting the origin for samples where prior martensite is present.**

The time shifts between transformation without martensite and with targets of 15 and 30% martensite are given in Table 2. Clearly, since the bainite formation kinetics are identical from the thus defined starting points, this shift amounts to the time that may be gained during the heat-treatment. In this matter, it is remarkable that forming 30% martensite prior to bainitizing at 220 C in 100CoCrSi10 allows a reduction in heat-treatment time of approx 45% and 36% (with respect to the times required to reach 90 and 95% transformation respectively).

The superimposition of these dilatometric signals suggests that target amounts of martensite were not achieved as expected, and were closer to 25 and 45% for targets of 15 and 30% respectively. This is presumably due to inaccuracy in the conversion from length change to transformed fraction but is without consequence for the discussion. In the following, the target amounts will continue to be used in the discussion.

**Table 2. Reduction in transformation time in hour, and as a percentage of time for 90% transformation.**

| **Bainitizing T** | **220°C** | | | **250°C** | | |
|---|---|---|---|---|---|---|
| **Target amount of martensite** | **0%** | **15%** | **30%** | **0%** | **15%** | **30%** |
| **100Cr6** | ref | 2.2 (29%) | 2.6 (34%) | ref | 1.0 (38%) | 1.3 (49%) |
| **100CoCrSi10** | ref | 3.3 (35%) | 4.2 (45%) | ref | 1.1 (36%) | 1.4 (45%) |

### Hardness and retained austenite measurements

Hardness measurements made on the dilatometric samples are given in Table 3, in addition, hardness in the as-quenched condition were 800 and 844 HV for 100Cr6 and 100CoCrSi10 respectively. The uncertainty estimated for all the present hardness measurements is about 3%, and it is therefore reasonable to suggest that there is no effect of prior martensite formation on the hardness achieved at the end of the bainite transformation.

**Table 3. Hardness measurements (HV)**

| **Bainitizing T / C** | **220** | | | **250** | | |
|---|---|---|---|---|---|---|
| **Target amount of martensite / %** | 0 | 15 | 30 | 0 | 15 | 30 |
| **100Cr6** | 746 | 739 | 732 | 696 | 697 | 690 |
| **100CoCrSi10** | 763 | 762 | 761 | 715 | 728 | 727 |

The retained austenite fraction was measured for the 100CoCrSi10 grade bainitized at 250 C with different volume fractions of martensite. Results are shown in Table 4. Here again, no effect of prior martensite formation is observed given the uncertainty associated with the measurements (typically 4%).

**Table 4.Retained austenite content (%).**

| Bainitizing T / C | 250 | | |
|---|---|---|---|
| Target amount of martensite / % | 0 | 15 | 30 |
| Residual austenite / % | 17.4 | 15.7 | 17.6 |

## Microstructure

Optical micrographs of 100CoCrSi10 bainitized at 250 C without prior martensite, and with a target of 30% of martensite, are presented at Figure 4. The 100CoCrSi10 exhibits a very fine bainitic microstructure after conventional bainitizing treatment at 250 C so that optical examination is not sufficient to distinguish martensite and bainite. Although scanning electron microscopy examination suggested the presence of distinguishable structures, their respective amounts was not correlated with the expected amounts of prior martensite, so that identification was not clear.

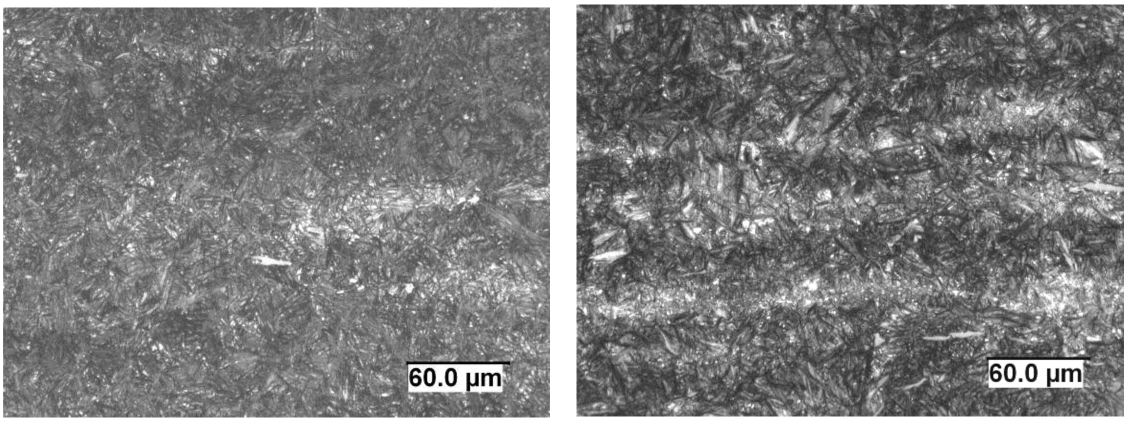


**Figure 4. Optical observations of 100CoCrSi10 bainitized at 250 C after Nital Etching wityhout martensite (left) and with 30% martensite (right).**

## Discussion

The effect of pre-quenching before isothermal treatment of bainitization on kinetics of bainitic transformation has been the subject of previous investigations [5-7]. The mechanisms leading to a reduction of bainitic transformation time due to prior martensite formation are still discussed. Kawata et al. [7] have shown that bainite transformation from an austenite and martensite dual-phase structure is faster than from single-phase austenite and the nucleation of bainitic ferrite laths are accelerated around martensite. They suggest that the martensite/austenite boundary acts as a nucleation site of bainitic ferrite since the effect on kinetics is the same in the case of pre-existing ferrite-pearlite. On the other hand, Vetters et al. [5] suggested that the initial pre-quenching favored later bainite formation because of shearing processes during martensite formation.

In the present study, the kinetics of low temperature bainite formation have been shown to be identical with or without prior martensite formation, in two high carbon grades (with and without Si). Indeed, whilst an overall reduction in heat-treatment time is clearly made possible with such a heat-treatment, the bainite reaction occurs, after formation of some fraction of martensite, in a virtually identical manner as it would if that first transformed fraction had been obtained through isothermal bainite reaction. This is consistent with an autocatalytic effect that is identical whether one considers the influence of existing martensite or bainite laths on subsequent bainite lath nucleation. In this sense, the results do not agree with those reported by Kawata et al. [7]: there is no detectable acceleration of the transformation since the rate with or without martensite is the same as expected at that level of progress in the austenite transformation.

Hardness measurements and optical examination of the samples with or without prior martensite formation did not reveal any significant differences. Although dual phase microstructures are sometimes said to be detrimental to fatigue resistance, this is related to significant heterogeneity in mechanical properties. In the present case, because retained austenite and hardness are left unchanged, it may be suggested that the tempered martensite and bainite present in the final microstructures have similar properties, so that a detrimental effect on fatigue resistance may not exist. This will be verified using rotation bending fatigue tests.

**Conclusions**

At temperatures slightly above Ms, the bainitic reaction kinetics in high carbon steels can be extremely sluggish. The influence of partial martensite formation on those kinetics have been investigated in two high carbon steels with and without Si, it is concluded that:

- bainitic heat treatment duration can be reduced by the formation of martensite before bainitic isothermal holding,
- the kinetics of bainite formation after prior formation of a given fraction of martensite are identical to those observed after formation of the same fraction of bainite,
- within the uncertainty of the measurements, final hardness and retained austenite content are not affected by the pre-existing martensite in the initial microstructure.

**References**

[1] C. Garcia-Mateo, F. G. Caballero and H. K. D. H. Bhadeshia, ISIJ Inter., 2003, Vol. 43, p1821-1825

[2] F. G. Caballero and H. K. D. H. Bhadeshia, Current Opinion in Solid State and Mater. Sci. 2004, Vol. 8, p251-257

[3] T. S. Wang, X. Y. Li, F. C. Zhang and Y. Z. Zheng, Mater. Sci. Eng. A, 2006, Vol. 438-440, p1124-1127

[4] X. Chen and E. Vuorinen, ISIJ Inter., 2009, Vol. 49, p1220-1224

[5] H. Vetters, J. Dong, H. Bomas, F. Hoffmann, H.W. Zoch, in International Journal Material Research, vol. 97, (2006), pp.1432-1440

[6] J. Dong, H. Vetters, F. Hoffmann, H.W. Zoch, in Journal of ASTM International, vol. 7, n°2, (2010)

[7] H. Kawata, K. Hayashi, N. Sugiura, N. Yoshinaga, M. Takahashi in Materials Science Forum, vols 638-642, (2010), pp.3307-3312

[8] D. P. Koistinen and R. E. Marburger, Acta Metall., 1959, Vol. 7, p59

[9] M. Onink, C. M. Brakman, F. D. Tichelaar, E. J. Mittemeijer and S. van der Zwaag, Scripta Metall. Mater., 1993, Vol. 29, 1011-1016