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INVESTIGATION OF AUSTENITE DECOMPOSITIONS IN HIGH-CARBON HIGH-STRENGTH FE-C-SI-MN STEEL UNDER 30 TESLA MAGENTIC FIELD

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Abstract

A 30-tesla magnetic field was applied during transformation of austenite to ferrite in two high-carbon high strength Fe-C-Si-Mn steels. Under normal cooling conditions, both of these steels transform to martensite due to retarded austenite transformation kinetics. The application of a 30-tesla magnetic field during continuous cooling at 1 °C/s resulted in a very fine pearlite with 50 to 100 nm lamellar spacing. Local electrode atom probe analyses of this pearlite showed fine-scale partitioning of substitutional elements. Isothermal transformation experiments at 740°C with and without an applied magnetic field showed similar austenite decomposition characteristics as that of continuous cooling conditions.

Introduction

Application of magnetic field to increase the driving force to austenite to ferrite transformation is a well-established concept [1]. Recently, the effect of 30-tesla magnetic field on phase transformations in a high-carbon bainitic alloys were evaluated [2] while continuously cooled at a rate of 1 °C/s. The results showed that in these alloys on continuous cooling without magnetic field lead to mostly martensitic microstructure with small amounts of bainite. In contrast, the same alloys with the presence of 30-tesla magnetic field transformed to fine pearlite. In this work, additional research has been performed to understand the alloying element partitioning characteristics during pearlite formation. Moreover, the tendency for the pearlite formation under isothermal conditions with and without magnetic field was evaluated.

Experimental Procedure

Alloys and heat-treatment

In this study two steels (see Table 1) with similar carbon concentration but different alloying element concentration were used. Both steels contain large silicon concentrations to retard the cementite precipitation during bainitic transformation. The steel identified as “SK” exhibits a very sluggish transformation kinetics. In contrast, the steel identified as “FK” is alloyed with Co and Al to enhance the transformation kinetics. These steels were homogenized for 48 h at 1200°C before subjecting to magnetic field processing.

Heat treatment and Magnetic Field Procedure

A 32mm diameter bore resistive magnet with a 33T maximum field strength at the National High Magnetic Field Laboratory (NHMFL) was used. An induction-heating coil set-up has been designed to heat and cool the specimen while inside the bore of the magnet. The apparatus locates the specimen in the center of the bore mid length and can heat the steel specimen up to 1100ºC and maintain the high temperature for extended periods of time. Atmosphere is controlled via an argon gas purge. For accelerated cooling, a helium gas quench can be rapidly imposed upon the specimen. Such a system allows for the entire thermal cycle or any portion of it to be exposed to a high magnetic field. Temperature measurements were made via a type "S" (Pt-10%Rh) thermocouple spot-welded on the surface at the mid-length of the specimen.

In this research, both SK and FK specimens were heated to 1000ºC using a two-step schedule where a rate of approximately 11ºC/s was applied to 700°C, followed by heating at 5ºC/s to the annealing temperature. A 3-minute hold at 1000ºC was performed to fully transform the initial microstructure to austenite. The samples austenitized at this temperature were subjected to three sets of experiments as summarized in Table 2.

Table 1. Compositions of steel used in the current investigation

Alloy	C	Si	Mn	Mo	Cr	Co	Al	V	Fe
Slow Kinetic Steel									
SK (wt.%)	0.75	1.63	1.95	0.28	1.48	-	0.01	0.10	Balance
SK (at.%)	3.34	3.10	1.89	0.16	1.52	-	0.02	0.10	Balance
Fast Kinetic Steel									
FK (wt.%)	0.78	1.60	2.02	0.24	1.01	3.87	1.37	-	Balance
FK (at.%)	3.43	3.01	1.94	0.13	1.03	3.47	2.68	-	Balance

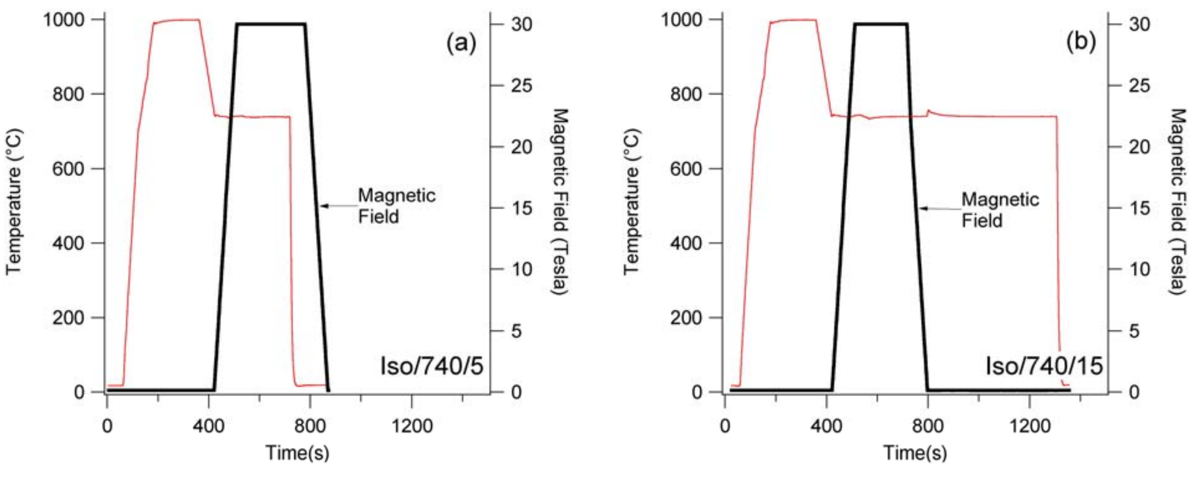

Fig. 1 Details of isothermal heat treatment procedure are shown with measured temperature and imposed magnetic field strength as a function of time. (*a*) First experiment where the 30-tesla magnetic field was applied after the samples reached the isothermal treatment temperature of 740°C and held at that level for the remaining heat treatment schedule including isothermal hold of 5 minutes and subsequent cooling. (*b*) Second experiment where the 30-tesla magnetic field was applied after the sample reached 740°C, however, after 5 minutes the magnetic field was reduced to 0.1 tesla for the remaining heat-treatment.

For continuous cooling with a magnetic field, the ramping of the field was initiated at one minute after reaching 1000ºC. A time of 1.5 minutes was required for the field to reach 30T. Specimen cooling was controlled via feedback loop such that by decreasing power to the induction coil, a 1ºC/s cooling rate was obtained. The temperatures of the samples were continuously monitored. In case of isothermal experiments two sets of experiments were performed. Typical temperature and magnetic field variations for the isothermal experiments are shown in Fig. 1.

In the first set, an effect of isothermal holding for 5 minutes at 740°C was compared with and without magnetic field in both the steels. The motivation for this experiment is simple comparison of isothermal transformation with and without magnetic field. This experiment is identified by “iso/740/5” keyword throughout the discussions.

In the second set, an effect of isothermal holding of 15 minutes at 740°C with magnetic cycling was adopted, i.e., magnetic field was switched on for 5 minutes and then switched off to for remainder of the heat treatment. The motivation for this experiment is to compare whether transformation that is accelerated for first 5 minutes with magnetic field will revert back for the remainder of 10 minutes when the magnetic field is switched off. This experiment is identified by “iso/740/15” keyword throughout the paper.

Table 2. Heat-treatment and magnetic field parameters used in the current investigation

Alloy/Heat Treatment	Isothermal Heat Treatment	Continuous Cooling Heat Treatment	Magnetic Field (Tesla
Continuous Cooling Experiment			
SK/CCT/NF	N/A	1 °C/s	No Field
SK/CCT/30T	N/A	1 °C/s	30
FK/CCT/NF	N/A	1 °C/s	No field
FK/CCT/30T	N/A	1 °C/s	30
Isothermal Experiments – Magnetic Field Effect			
SK/iso740/05/NF	740°C/5min	N/A	No Field
SK/iso740/05/30T	740°C/5 min	N/A	30
FK/iso740/05/NF (F)	740°C/5min	N/A	No Field
FK/iso740/05/30T (H)	740°C/5 min	N/A	30
Isothermal Experiments –Magnetic Cycling			
SK/iso740/15/NF	740°C/15min	N/A	No Field
SK/iso740/15/30T	740°C/15 min	N/A	30 (5 min) & 0.1 (10 min)
FK/ISO740/15/NF	740°C/15 min	N/A	No Field
FK/iso740/15/30T	740°C/15 min	N/A	30 (5 min) & 0.1 (10 min)

Characterization

The sample that exhibited very fine scale microstructure was characterized with transmission electron microscopy and local electrode atom probe technique. The samples for this characterization were prepared using standard electro polishing technique. A 200 kV Tecnai-20 transmission electron microscope was used to characterize thin foils and to characterize the fine scale precipitate composition; the Oak Ridge National Laboratory local electrode atom probe was used. A specimen temperature of 60 K, a pulse repetition rate of 200 kHz and a pulse fraction of 20% were used for the analyses. Compared to previous types of atom probes, this new instrument has a significantly faster rate of data acquisition and a larger field of view and hence larger volumes of analysis.

Results and Discussions

Transformation during continuous cooling

Some of the results from the continuous cooling experiment have been presented already published [2]. However, to compare with the isothermal experiment, they are briefly summarized. The SK and FK steels subjected to continuous cooling from austenitizing temperature without magnetic field did not show any major differences in the microstructure [see Fig. 2*a* and 2*b*]. On application of a 30-tesla magnetic field, the SK steel showed some localized regions of softer microstructure [Fig. 1*c*]. In contrast, the FK steel with an applied magnetic field led to a uniform soft microstructure [see Fig. 1*d*]. Since this fine microstructure could not be resolved in optical microscopy, transmission electron microscopy and local atom probe microanalysis were performed.

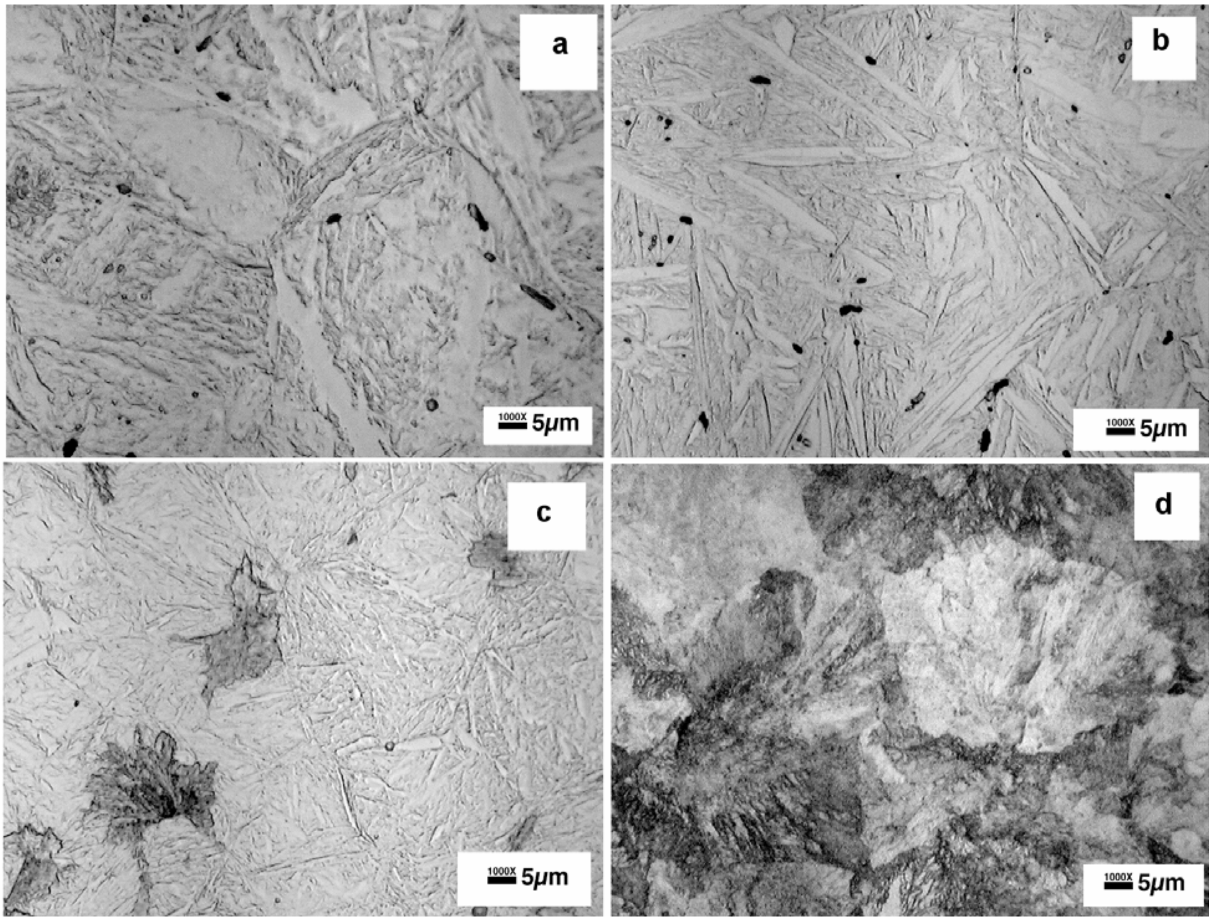

Fig. 2 Summary of microstructures attained by continuous cooling from austenitizing temperature with and without magnetic field: (*a*) SK steel – No field; (*b*) FK steel – No field; (*c*) SK steel – 30 Tesla; and (*d*) FK steel – 30 Tesla.

Transmission electron microscopy [see Fig. 3] of FK steel subjected to 30 Tesla showed that the fine microstructure is pearlite with lamellar spacing in the order of 50 nm. The local electrode atom probe analysis of the same sample is presented in Fig. 4. The iso-concentration surface corresponding to 15 at. % carbon outlines a small cementite plate in Fig. 4*a*. The above surface shows that the cementite plate thickness is less than 10 nm. The atom images in Fig. 4*b* show preferential partitioning of Cr, Mo and Mn to the cementite. In addition, a concentration profile of all elements was calculated perpendicular to the cementite plate and is shown in Fig. 4*c*. The concentration profile clearly shows the partitioning of elements between ferrite and cementite has taken place, i.e., the elements like Cr and Mn partition to the cementite, while the elements like Al, Si, and Co partition to the ferrite. The partitioning of Mo to cementite is indeed present, however, less strong than Cr and Mn. The measured partitioning agrees with the trends predicted by thermodynamic calculations. It is important to note that this result clearly shows the decomposition of the austenite into pearlite in the presence of magnetic field occurs by reconstructive mechanisms. Although, the electron diffraction and atom probe analysis did not show any other form of alloy carbides in the pearlitic microstructure shown in Fig. 3, further detailed characterization using extraction technique is needed to confirm the absence of other alloy carbides.

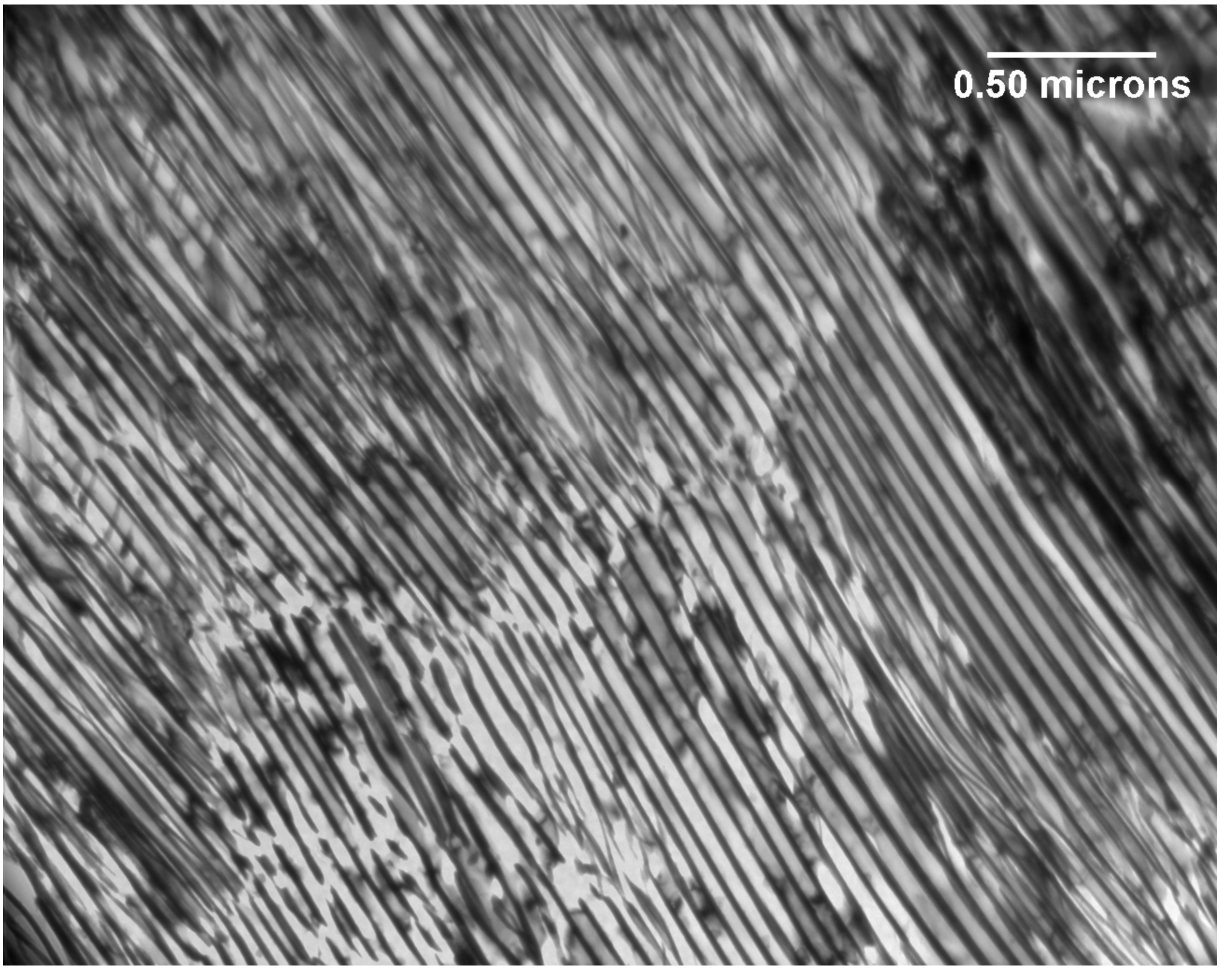

Fig. 3. Transmission electron micrograph from FK steel subjected to continuous cooling from austenite phase field at a rate of 1 °C/s with the presence of 30-tesla magnetic field showing the presence of fine pearlite.

A reconstructive mechanism for pearlite formation is based on rigorous atomic motion across the austenite/ferrite/cementite interfaces, which facilitates the preferential partitioning of substitutional elements between ferrite and cementite. In agreement, the measured temperature records during this transformation showed a recalescence effect at 700°C [2] at which the substitutional atom mobility is still sufficient enough to allow for reconstructive transformation. The current results are in agreement with earlier atom probe results from pearlite [3]. In addition, other works have shown that the pearlite always exhibits substitutional partitioning, even though the extent did not reach the expected equilibrium level [4,5,6,7]. It is important to compare this result with the formation of cementite during martensite tempering. Previous work have shown that the fine cementite which forms during martensite tempering does not show any preferential partitioning of any substitutional elements including silicon which has zero solubility within cementite [8,9].

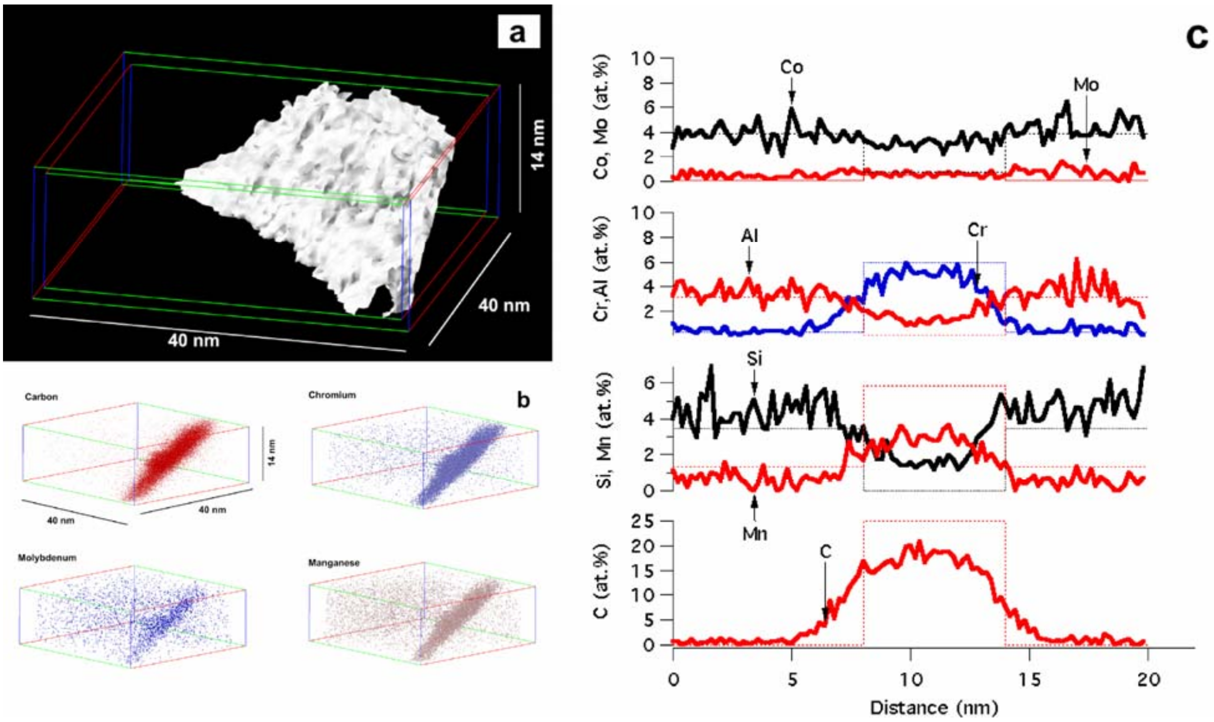

Fig. 4. Summary of local electrode atom probe microanalysis result from the FK/CCT/30T sample. (*a*) 15 at.% carbon iso-concentration surface showing the morphology of cementite lamellae. (*b*) Cr, Mo, and Mn atom maps showing the preferential partitioning to the C-enriched cementite region. (*c*) Concentration profile (10 nm X 10 nm cross section) perpendicular to the cementite plate showing the preferential partitioning of Cr, Mn, Mo to cementite and Al, Si and Co to ferrite. The dotted lines show the predicted alloying element concentration between ferrite and cementite at 740°C by thermodynamic calculations [10,11].

<u>Transformation during isothermal transformation</u>

In order to evaluate the tendency for the pearlite formation with and without magnetic field, both the alloys were subjected to isothermal heat treatment. The results from the SK steel are summarized in Fig. 5. Without the application of magnetic field, the isothermal transformation for either 5 or 15 minutes at 740°C [see Fig. 5*a* and 5*c*] did not lead to any pearlitic transformation. The austenite essentially transformed to martensitic microstructure. However, on application of 30-tesla magnetic field [see Fig. 5*b* and 5*d*], the austenite transformed to pearlitic microstructure as the sample was held for 5 minutes and 15 minutes. The transformation to pearlite appears to be complete after 5 minutes. As a result, in the 740/15/30T experiment, there was no significant microstructural difference even though the magnetic field strength was reduced to 0.1 during the remainder of the experiment.

The results from FK steel, which contains increased levels of Al and Co that enhances the austenite decomposition kinetics, are shown in Fig. 6. In contrast to SK steel, the FK steel showed some differences even without magnetic field. In this steel after isothermal hold of 5 minutes at 740°C [see Fig. 6*a*], pearlite formation was not observed. However, after isothermal hold for 15 minutes at 740°C, this steel transformed to coarse pearlitic microstructure [see Fig. 6*c*]. The application of 30-tesla magnetic field induced the pearlite formation [see Fig. 6*b* and 6*d*]. Similar to SK steels, the transformation to pearlite appears to be complete after 5 minutes and there was no significant microstructural difference between 5 and 15 minutes hold times.

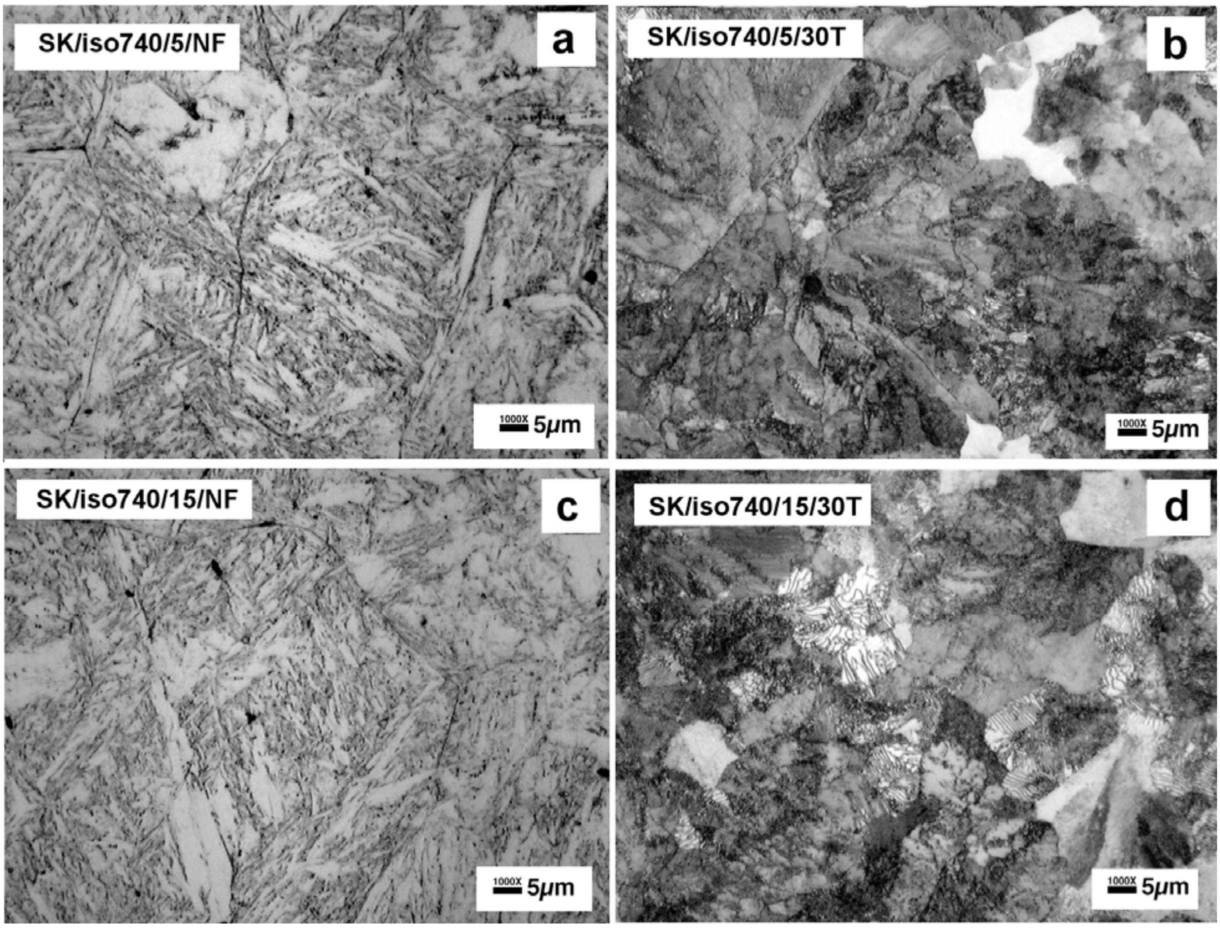

Fig. 5 Summary of microstructures attained by isothermal treatment of SK steel at 740°C with and without magnetic field: (*a*) no field and 5 min; (*b*) 30-tesla field and 5 min; (*c*) no field and 15 min and (*d*) 30-tesla field for 5 minutes and 10 min with 0.1 Tesla.

The above results clearly show the effect of magnetic field does accelerate the transformation in both steels even under isothermal transformation conditions. In addition, the comparison of SK and FK steels shows there is a synergistic effect between alloying elements such as aluminum and cobalt and magnetic field.

Thermodynamic Calculations

To evaluate the effects of magnetic field on the stability of various phases in SK and FK steels, thermodynamic calculations were performed using ThermoCalc software [10] and ThermoTech Fe database [11]. In the next step, the free energy of ferrite is decreased in order to simulate the effect of the magnetic field in stabilizing ferrite and the effect on cementite and other phases is neglected. The reduction of free energy of ferrite per Tesla of magnetic field is calculated to be 12 J/mole. For a 30-T magnetic field this amounts to 360 J/mole. The phase stabilities of both alloys are compared at 770°C and 740°C. The results are shown in Fig. 7.

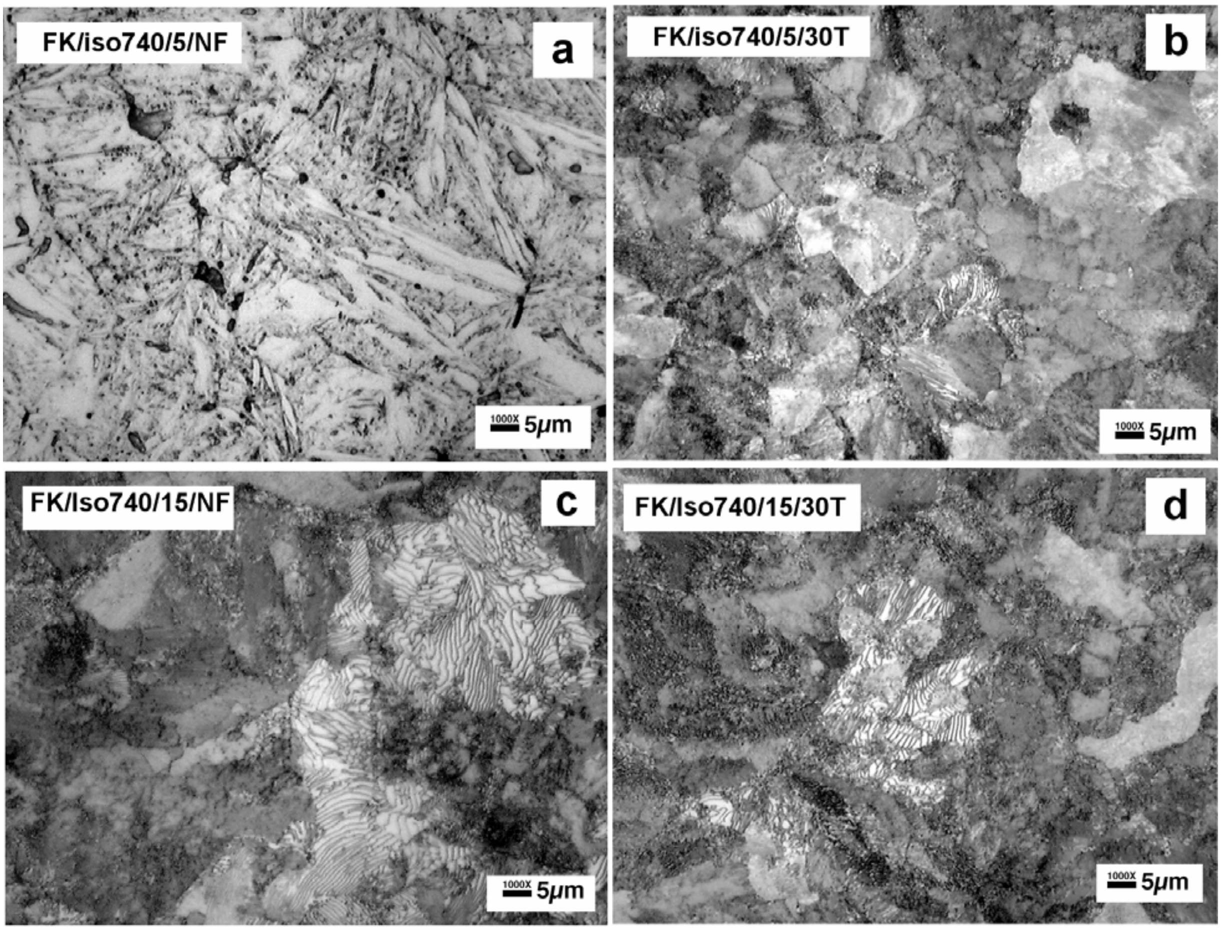

Fig. 6 Summary of microstructures attained by isothermal treatment of FK steel at 740°C with and without magnetic field: (*a*) no field and 5 min; (*b*) 30-tesla field and 5 min; (*c*) no field and 15 min and (*d*) 30-tesla field for 5 minutes and 10 min with 0.1 Tesla.

The calculations show that for FK steel at 770°C† the most stable phase is austenite without the presence of magnetic field. As expected, with the presence of magnetic field the stable phase becomes ferrite and cementite. Therefore, this diagram shows the effect of magnetic field on changing the phase stability of this steel. In contrast, the phase stabilities of SK steel at 740°C, FK steel at 770°C and 740°C do not show a large difference and suggest that the most stable phases are ferrite and cementite. This indicates that if we isothermally held these two steels at 740°C, the most stable phase would be ferrite and cementite with or without magnetic field. These predictions need to be compared with the observed microstructure in both steels. In SK steels, even though the most stable phase at 740°C, is ferrite and cementite, the transformation of austenite to pearlite did not occur. This is attributed to the sluggish transformation kinetics in these steels. Similar to SK steel, FK steel did not transform to pearlite on holding at 740°C for 5 minutes. Surprisingly, the same steel transformed to pearlite after holding for 15 minutes without magnetic field. This phenomenon is attributed to the acceleration of pearlite kinetics due to the addition of Co and Al. Therefore, the results from FK steel show that the magnetic field also has an effect on the kinetics. However, the current data cannot be used to deconvolute whether this acceleration was achieved through acceleration of either nucleation or growth or both. There is a need to measure the transformation kinetics quantitatively with the presence of magnetic field.

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† 770°C is the curie point of ferrite in pure iron.

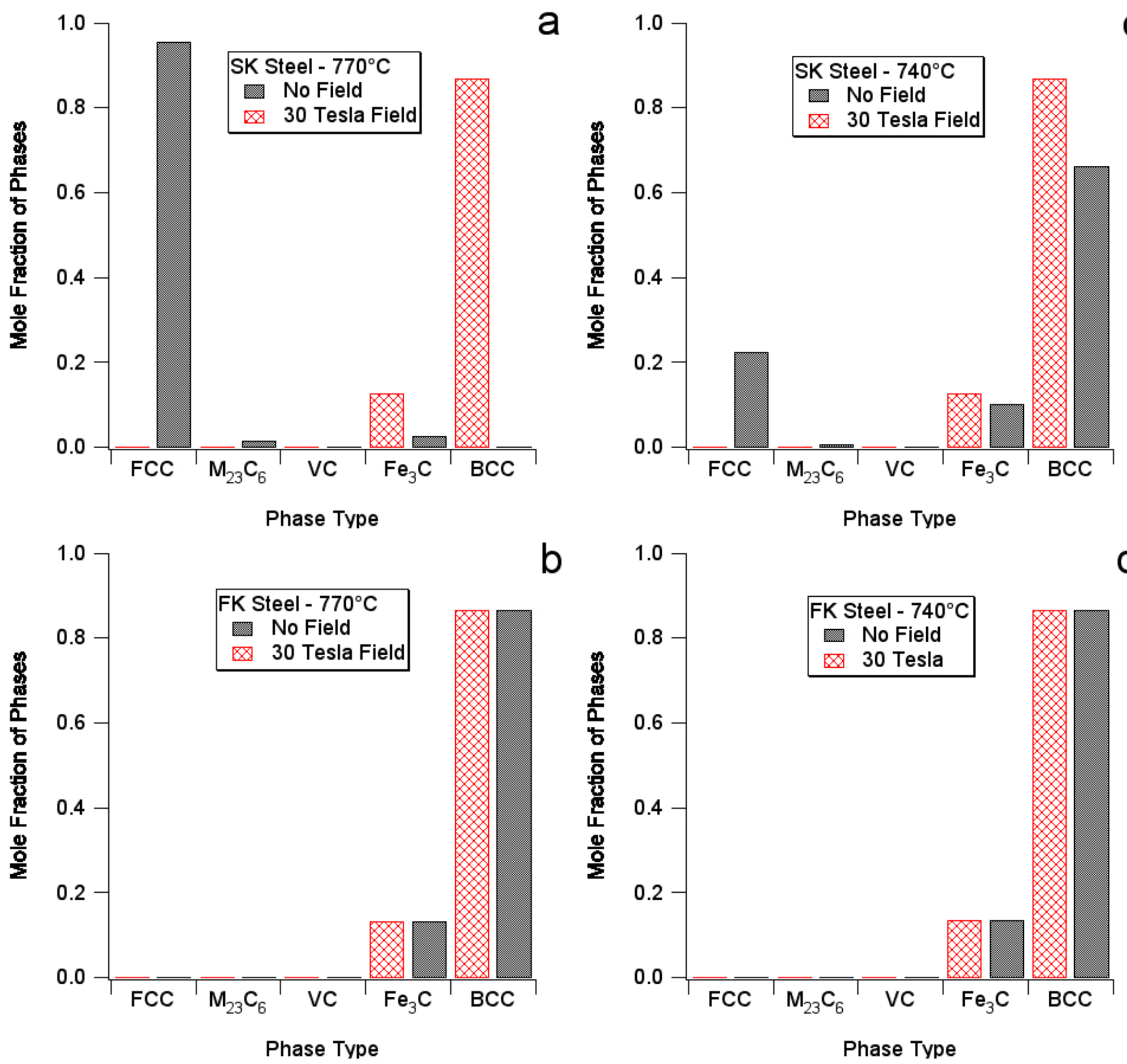

Fig. 7. Summary of phase stability calculations with and without consideration of free energy contribution to ferrite due to 30 T magnetic field: (*a*) SK steel T=770°C; (*b*) SK Steel T=740°C; (*c*) FK steel T=770°C; and (*d*) FK Steel T=740°C.

Summary and Conclusions

Effects of 30-tesla magnetic field on the austenite decomposition in two high-carbon steels with different austenite decomposition kinetics were investigated. In the steel alloyed with Al and Co, the decomposition of austenite to fine pearlite occurred while continuous cooling from austenite phase field. The pearlite microstructure exhibited a lamellar spacing of less than 50 nm. Local electrode atom probe analysis of this pearlite microstructure showed enrichment of Cr, Mn, Mo and C and depletion of Si, Al and Co in the cementite phase. The decomposition of austenite without addition of Al and Co was not extensive during continuous cooling, due to the sluggish kinetics.

The effect of the magnetic field was also evaluated under isothermal transformation conditions at 740°C. In the steel with addition of Al and Co, the pearlite formed with and without magnetic field; however, the magnetic field accelerated the kinetics of transformation. In case of steel without addition of Al and Co, the transformation to pearlite occurred only with the presence of magnetic field. The results also showed in both the steels, after the completion of the transformation of austenite to pearlite, removal of magnetic field did not lead to any change in the microstructure. Thermodynamic calculations indicated that at 740°C, both the steels should transform to pearlite. Therefore, the present results indicate that the magnetic field accelerated the transformation kinetics in synergy with addition of alloying elements including aluminum and cobalt.

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