

Boron-Based Carbon Fiber Coating Of low Carbon Steel for Ultra-Fast Surface Hardening

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Abstract

Ultra-fast surface hardening of low carbon steel is introduced via the application powder-pack boriding process in a hot isostatic pressing special fixture. Boriding (Boronizing) powder consisting of boric acid and borax mixture is utilized in 50 wt. % boric acid and 50 wt. % of borax. Low carbon steel sample packed with boric acid and borax, heated at 1050°C for 30 minutes and then at tempered 900°C for 30 minutes in a special fixture sealed with a 10 Ton pressure. The surface boride layer as FeB and Fe₂B phase with the hardness about ~1800 HV and depth of 130-180 μm is achieved and compared to untreated base metal of 123 HV. Alloy segregation along with delocalized zone of interest is achieved with different boron content 1.77 wt. % along grain boundaries, 3.93 wt. % leading phase and 7.86 wt. % trailing phase at the sample surface. This process provides high performance and high thickness of coatings and can be used fast and economically if compared to traditional processes with less emission. From economic and environmental points of views, it is highly desirable to develop and implement novel surface treatment technologies that are quick, cheap, clean, and energy efficient.

Introduction

High-temperature treatment processes (such as nitriding, carburizing, and boronizing) are used widely to achieve superior mechanical and tribological properties on ferrous materials [1-3]. However, these traditional processes are very slow and energy intensive, producing large amounts of CO₂ emissions and liquid/solid wastes. Conventional boriding processes such as pack, paste, plasma, fluidized bed, and salt-bath boriding are, environmentally unsafe, and energy intensive; in addition, they are slow and limited in their ability to create a dense and thick boride layer [1]. It requires several steps for a successful boriding run and takes more than 6 hours to produce a 45-μm-thick boride layer on a low-carbon steel part.

The most common boron-yielding or releasing powders used in pack boriding include boron carbide (B₄C), ferroboration, and amorphous boron [2]. The powder mixture also uses a variety of diluents such as silicon carbide (SiC) and aluminum oxide (Al₂O₃) powders, as well as numerous catalyzers or boron activators such as sodium fluoroborate (NaBF₄), potassium fluoroborate (KBF₄), ammonium fluoroborate [(NH₄)₃BF₄], ammonium chloride (NH₄Cl), sodium carbonate (Na₂CO₃), and barium fluoride (BaF₂) [3-4]. Traditional pack boriding is typically done in sealed or packed retorts at very high temperatures. It has large amounts (about 90%) of boron carrier powder materials (like SiC), a source of boron (like boron carbide or amorphous boron), and an activator (like KBF₄) to enhance diffusion of atomic boron into the work piece [1, 2, 5]. Pack boriding involves placing the work pieces into the powder mix and sealing them in a container. The

container is then heated up to around 950°C and held there for 4 to 8 hours and finally cooled in air [6-7]. The thickness of the boride layers is determined by the process temperature and duration but typically is in the range of 50 to 100 µm. Powders used in the pack pose difficult waste management and disposal problems and are not easy to recycle or reuse. In the case of salt-bath boriding, a molten salt bath consisting of 60 to 70 wt. % borax, 10 to 15 wt. % boric acid, and 10-20 wt% ferrosilicon or ferroboration is used at temperatures ranging from 900 to 1000°C. Boride layers forming on the surface are typically 10-15 µm thick after 7 to 10 hours of treatment [6].

A novel surface treatment technologies that are quick, cheap, clean, and energy efficient is highly desirable to develop and implement. Ultra-fast boriding process is introduced to achieve thick, hard, and uniform boride layers on low carbon steel components. Thick boride layers are attained on steel components consisted of FeB, Fe₂B, and phases, and compared to slow processes that gain hardness values ranged from 16 to 22 GPa [7]. Much improved mechanical properties enabled by ultra-fast boriding could substantially increase product life, which indirectly reduces cost and energy consumption due to the elimination of the frequent repair and/or re-manufacturing of failed parts. For comparison, the mechanical hardness of a typical nitrided and/or carburized surface is in the range of 6.5 to 11 GPa [4]. Ultra-fast boriding process is established at 1050°C for 30 minutes, tempered at 900°C for 30 minutes in a special fixture sealed with a 10 Ton pressure. The kinetic of powder pack at high temperature 1050°C along with tempering at 900 °C produce thick borided layer (130-180 µm) with high boron and carbon content at coating surface (7.86 wt. % B, and 4.14 wt. % C). Alloy segregation is more feasible with tempering temperature induces segregation of boron, and carbon atoms not only along the leading phase and surface of coating layer but also inter-granular bulk treated matrix.

Results and Discussion

Test samples of cylindrical shape low carbon steel (AISI 1020) with dimension 25 mm diameter and 5 mm thickness are prepared. A mixture of 50% borax and 50% boric acid are used in a powder packing the sample in a special fixture under a press of 10 ton, then heated at 1050 °C for 15 to 30 minute with tempering at 900°C for 30 minutes. The application of hot isostatic press (HIP) of a low carbon steel sample in a laboratory scale is verified. The applied pressure induces a complete tightness, complete mixture contact to the sample and air bleeding. It is a one step reaction that produces a thick borided layer of 130 µm to 180 µm in short time 30 minute. Optical microscopy and SEM microscope studies showed that the borides formed on the AISI 1020 steel have columnar nature. The presence of FeB and Fe₂B boride was determined by EDX analysis.

Figure 1 presents optical microscopy of test sample at 1050°C for 15 minute holding time and air cooling under press of 10 Ton. Boriding layer of thickness around 20 µm is established, with minor diffusion revealed within bulk treated matrix and presented in Figure 2. Scanning electron microscopy of test sample HIPed at 1050°C for 30 minutes reveals growth in boriding layer and presented in low magnification (Figure 3), and high magnification (Figure 4). Figure 5 presents scanning electron microscopy of leading phase and treated matrix in test sample HIPed at 1050°C for 30 minute at high magnification. It illustrates leading phases in boriding layer as well as inter-granular diffused phases that have a great effect on treated matrix as correlated with optical microscopy of a treated sample (Figure 2). Figure 6 presents scanning electron microscopy of bulk matrix next to boriding layer in a test sample HIPed at 1050°C for 30 minute. Figures 2 to 5 reveals delocalized dark zones along coating layer as well as bulk matrix. The addition of a tempering process at 900°C is introduces with more alloy segregation along coating surface as well as bulk matrix. Figure 7 presents SEM of leading phase in boriding layer of test sample HIPed at 1050°C for 30 minute and tempered at 900 °C for 30 minute. More alloy segregation introduces

with leading columnar interface with delocalized dark areas along surface as well as within bulk matrix [Figures 7 and 8].

The kinetic of boriding in powder pack coating at 1050°C for 30 minute with tempering at 900 °C for 30 minute induce segregation of alloying elements (B, C, and Fe). Figure 8 presents EDX of borided and tempered sample at three locations (A, B and C) for outer surface, leading borided columnar structure, and treated area respectively and presented in Figures 9, 10 and 11. Micro-hardness is applied on the boriding layer [Figure 11] and presented in Table 1. The hardness correlated with high boron content along coating surface (7.86 wt. % B) that relates to FeB layer thickness with Vickers hardness 1893 Hv. Leading interface Fe₂B with boron content 3.93 wt. % B produces hardness 441 Hv. Treated bulk matrix next to interface has 1.77 wt. % B with 226 Hv compared to untreated surface hardness of 123 Hv. In addition to boron, a new element (carbon) is introduced along borided surface (4.14 wt. % C), at the leading interface (3.77 wt. % C), and at treated matrix (2.98 wt. % C). Ultra-fast boriding with tempering not only introduce boriding layer enriched with boron and carbon but also introduce the two elements to the bulk treated matrix if compared to untreated matrix [Table 1]. The surface boride layer as FeB (at the surface) and Fe₂B (leading columnar phase) with surface hardness about ~1800 HV and depth of 130-180 μm is achieved via ultra-fast boriding.

Conclusions

The application of powder pack at high temperature 1050°C is established with clear boriding thickness of 20 μm at 20 minute holding time. A mixture of boron carrying powder (50wt. % borax, and 50 wt.% boric acid) is melted and sealed under 10 Ton press has a great effect on fast boriding. Holding time can also accelerate boriding process introducing thicker layer of 130-180 μm depth. Inter granular diffusion of boron is established in bulk treated matrix that enhanced its hardness compared to untreated matrix. In addition delocalized carbon enriched zones are introduced not only along coating layer but also within treated bulk matrix. The segregation of alloying elements is more dominant resolving variety of boron, and carbon content along surface, leading interface and treated bulk matrix.

The effect of tempering process along with ultra-fast boriding has a great effect on introducing surface boride layer as FeB and Fe₂B phase with the surface hardness about ~1800 HV and depth of 130-180 μm. The obtained hardness value is more than ten times if compared with the untreated specimen that only had the hardness of 123 HV. Boriding temperature, holding time, and powder-pack boriding pressure, along with tempering temperature and time has a great effect on ultra-fast boriding and surface hardening leading to a diffusion controlled process. This process provides high performance and high thickness of coatings and can be used fast and economically if compared to traditional processes with less emission. From economic and environmental points of views, it is highly desirable to develop and implement novel surface treatment technologies that are quick, cheap, clean, and energy efficient.

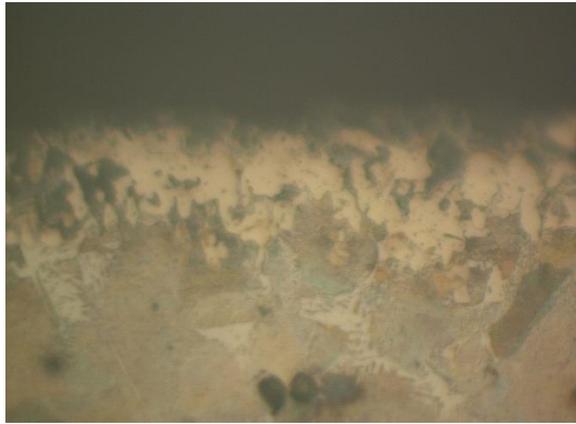


Fig. 1. Optical microscopy of test sample HIPed at 1050°C for 15 minute holding time and air cooled under press of 10 Ton.



Fig. 2. Optical microscopy of test sample HIPed at 1050°C for 15 minutes, air cooled under 10 Ton press.

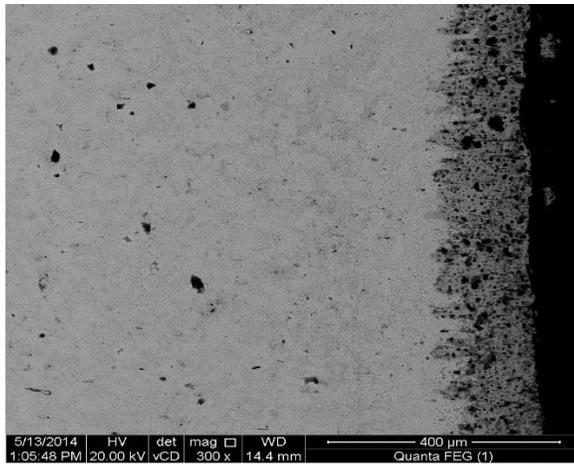


Fig. 3. Scanning Electron Microscopy of test sample HIPed at 1050°C for 30 minute and air cooled under 10 Ton press. [Low Mag.]

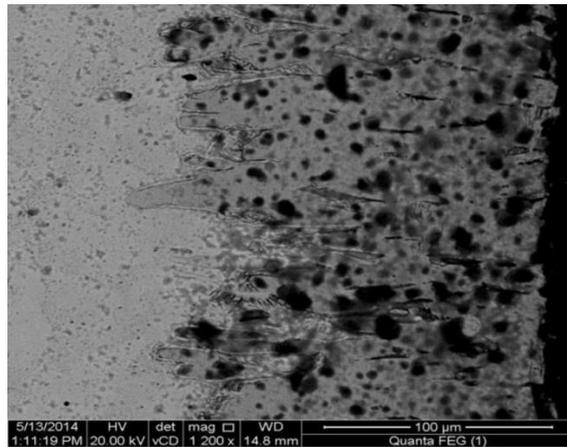


Fig. 4. Scanning Electron Microscopy of test sample HIPed at 1050°C for 30 minute, and air cooled under 10 Ton press [High Mag.]

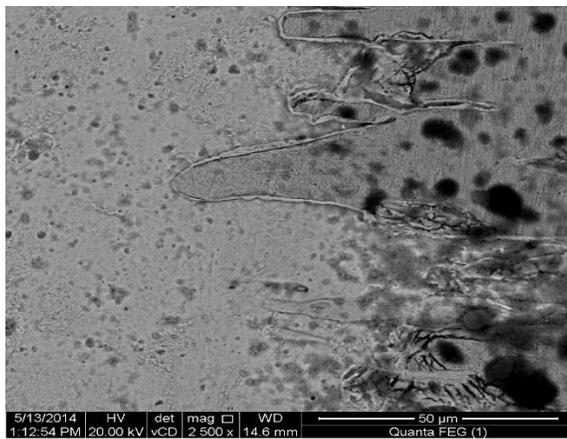


Fig. 5. Scanning Electron Microscopy of leading phase in bordering layer of test sample HIPed at 1050°C for 30 minute. [High Mag.]

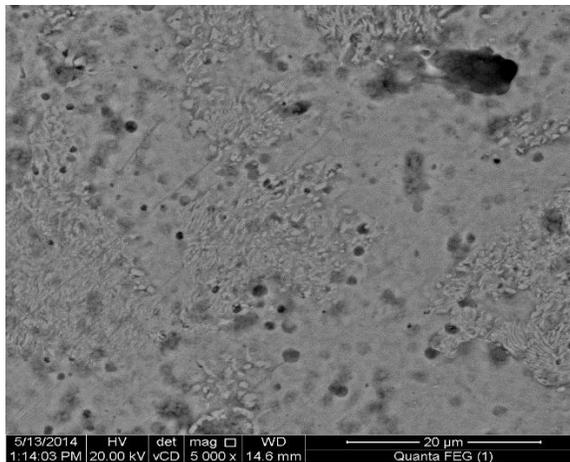


Fig. 6. Scanning Electron Microscopy of treated bulk matrix in a test sample HIPed at 1050°C for 30 minute. [High Mag.]

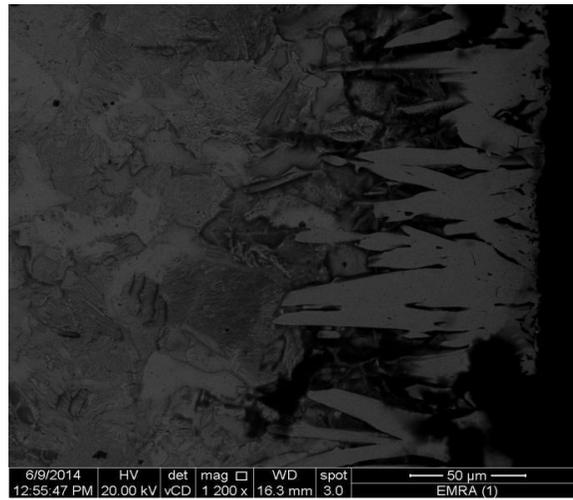


Fig. 7. SEM of leading phase in boriding layer with matrix constituents of test sample HIPed at 1050°C for 30 minute, and tempered at 900°C for 30 minute.

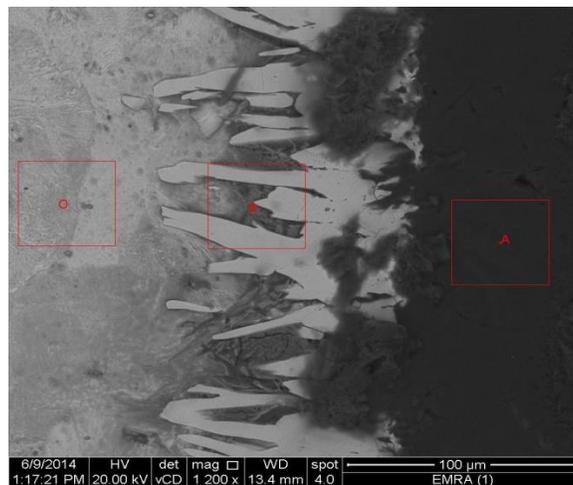


Fig. 8. SEM with three site of EDX of test sample HIPed at 1050°C for 30 minute and tempered at 900°C for 30 minute.

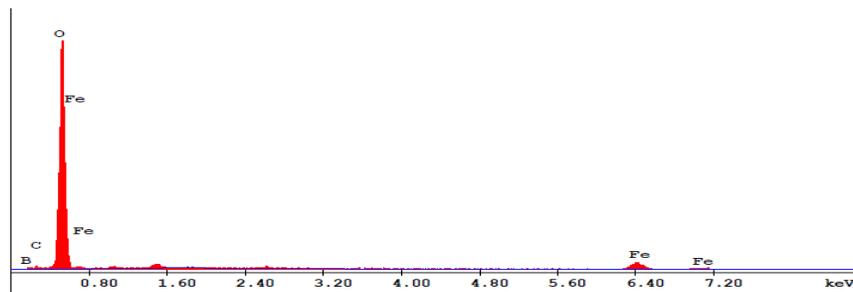


Fig. 9. EDX of boriding layer (A-Outer Surface) of test sample HIPed at 1050°C for 30 minute.

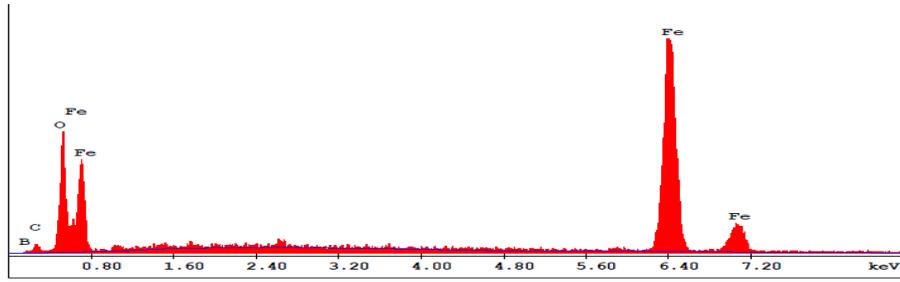


Fig. 10. EDX of boriding layer (B-leading boriding phase) of test sample HIPed at 1050°C for 30 minute.

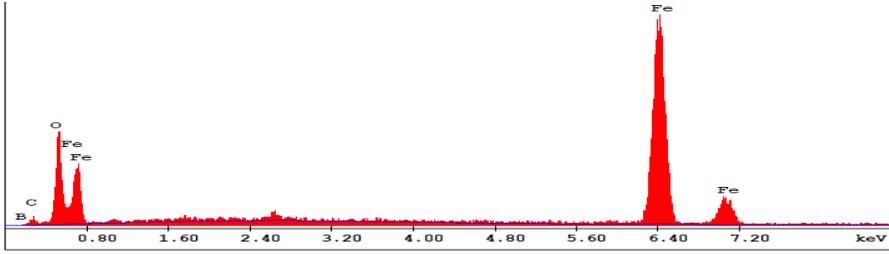


Fig. 10. EDX of boriding layer (C-treated Bulk matrix materials) of test sample HIPed at 1050°C for 30 minute.

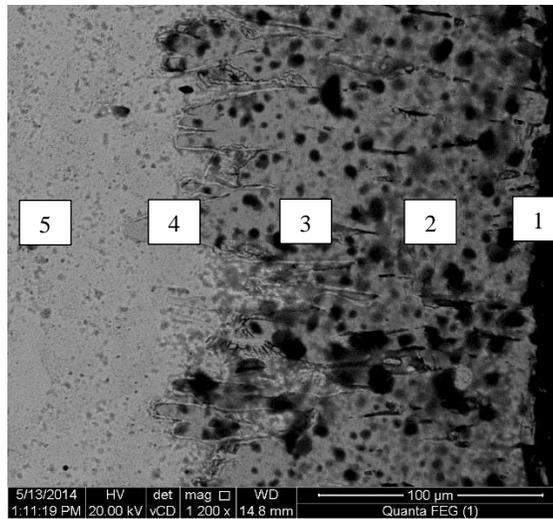


Fig. 11. Micro-hardness test site at coating surface and bulk treated and untreated matrix.

Table. 1 Micro hardness at different site of boriding sample.

point	Micro-hardness [HV]	Identification	Boriding elements	
			Wt. % B	Wt. % C
1	1893	Outer coating layer	7.86	4.14
2	1649	Inner coating layer	6.90	4.03
3	1028	Inner coating layer	5.22	3.89
4	441	Leading phase - front	3.94	3.77
5	226	treated matrix-bulk	1.77	2.98
As received	123	Untreated matrix-bulk	-	-

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