

Issue - January 2017

1. Recent Vanadium Related Publications Review
2. Recent Vanadium Related Publication List
3. Calendar of Technical Conferences and Seminars
4. Vanitec Members

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Vanitec is a technical and scientific committee, which brings together representatives of companies and organisations involved in the mining, processing, manufacture, research and use of vanadium and vanadium-containing products.

Vanitec establishes, collects and makes available information on vanadium, its use, availability, new scientific and technical developments and trends in its application. The object of V-Technology is to provide up to date information and to initiate discussion through the medium of short articles on subjects to the use of vanadium.

Recent Vanadium Related Publications Review

Vanadium Increases Bendability and Hole Expansion Coefficient of Cold-rolled Martensitic Ultra-high Strength Steel for Roll Forming

Martensitic ultra-high strength automotive steels are usually formed using the energy intensive hot stamping for car body applications. However, over the past decade, cold roll forming has experienced rapid development for shaping martensitic ultra-high-strength automotive steels. The roll forming process is an incremental bending process that turns a flat sheet into a structural profile as compared to traditional stamping process that involves severe stretching of the sheet to create the required part geometry. Therefore, cold roll forming only requires the steel to be bendable and it is suitable for forming martensitic ultra-high strength steel strip products, which typically exhibit high bendability, but have relatively low total elongation and poor formability for traditional stamping technologies. In addition, the cold forming process has also relatively low cost and high production efficiency compared to the traditional hot stamping.

As the automotive industry continues to demand materials exhibiting higher strength in the final formed part, reducing costs associated with both the material and the forming technology are also required. Therefore, development of martensitic ultra-high-strength steels for roll forming technologies has become a priority topic in the steel industry. T. Taylor of Tata Steel Strip Products UK recently published a paper entitled "Novel Cold-rolled Martensitic Ultra-high Strength Steels for Roll Forming Technologies" in Materials Science and Technology, Feb. 2016, pages 1-22. The paper showed the development of the steel chemistries and continuous annealing processes at Tata Steel Strip Products UK to produce low alloy cold rolled continuously annealed martensitic ultra-high-strength steel strip products for automotive cold roll forming technologies. Six laboratory produced experimental steels, differentiated by carbon content and independent additions of molybdenum, vanadium and nickel were used in the study and the chemical composition of steels are shown in Table 1. The experimental steels were laboratory produced by Tata Steel Research & Development UK. The cast ingots were hot rolled to sheet with a thickness of 2 mm via seven passes and then the hot rolled sheet were cold rolled into thickness of 1.5 mm. The steels were subjected to laboratory continuous annealing simulations with three investigated soak temperatures of 800°C, 850°C and 900°C. The optimal soak temperature, which is defined by that giving rise to the highest ultimate tensile strength-total elongation product, was selected to be 900°C for Steel B, Steel B+Mo and Steel B+Ni and 850°C for Steel B+V, Steel B+C1 and Steel B+C2. The bend test and hole-expansion capacity (HEC) test were carried out on the specimens with the optimal soak temperature conditions.

The final martensitic ultra-high strength steel sheets exhibit ultimate tensile strength from 1600 to 2200 MPa, total elongation from 3% to 11%, uniform elongation from 2-6, bend ratio from 5t to 2t and hole-expansion capacity from 18% to 80% for the optimal soak temperature conditions. Increasing carbon content from Steel B to Steel B+C1 and to Steel B+C2 generally give rise to higher strength, but compromise formability properties.

Table 1 Chemical composition of the steels (wt.%)

| Steel | C | Mn | Si | Other | CEN |
|------------|-------|-------|-------|---------------|-------|
| Steel B | 0.245 | 1.195 | 0.178 | Cr, Ti, B | 0.517 |
| Steel B+Mo | 0.257 | 1.180 | 0.202 | Mo, Cr, Ti, B | 0.581 |
| Steel B+V | 0.255 | 1.200 | 0.190 | V, Cr, Ti, B | 0.553 |
| Steel B+Ni | 0.255 | 1.310 | 0.190 | Ni, Cr, Ti, B | 0.561 |
| Steel B+C1 | 0.291 | 1.260 | 0.179 | Cr, Ti, B | 0.565 |
| Steel B+C2 | 0.380 | 1.200 | 0.190 | Cr, Ti, B | 0.660 |

Each alloy addition of molybdenum, vanadium and nickel to the 'base' chemistry (Steel B) gave rise to higher strength and increased the total elongation as shown in figure 1. These molybdenum, vanadium and nickel added steels also showed the very respectable ultimate tensile strength-total elongation products of more than 15000 MPa%.

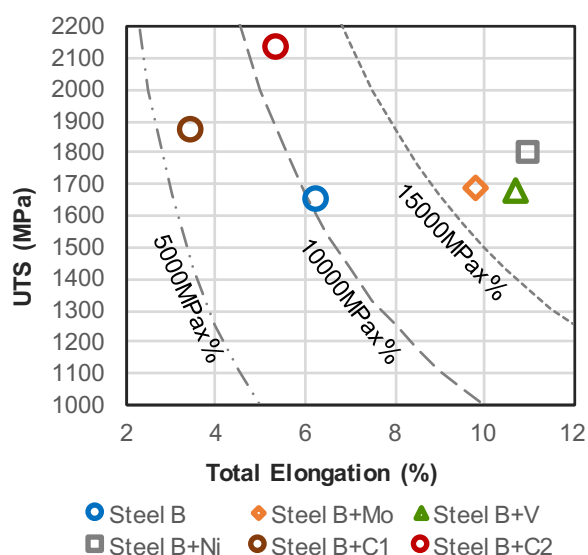


Fig. 1 Ultimate tensile strength against total elongation

There was a consistent positive correlation between uniform elongation and bendability and a near consistent positive correlation between uniform elongation and HEC, as shown in figure 2, indicating that bendability and

HEC are dependent on uniform elongation. There was also a near consistent positive correlation between bendability and HEC. The molybdenum and vanadium additions increased uniform elongation and bendability, while the molybdenum, vanadium and nickel additions all increased the HEC. The vanadium added steel (B+V) exhibited the highest HEC of more than 80% (Figure 2).

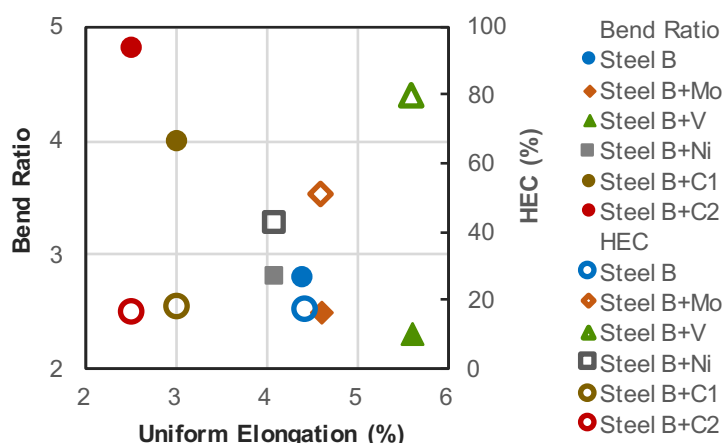


Fig. 2 Vanadium improves ductility, bendability and formability

It was concluded that the developed steels exhibit very attractive properties for roll forming technologies and provide low cost down-gauging opportunities with improved crashworthiness for the automotive industry.

Vanadium Improves Very High Cycle Fatigue Properties of Cr-Mo Low Alloy Steel

In recent years, very high cycle fatigue (VHCF) constitutes one of the main design criteria for many mechanical structures such as railway wheels, rails, offshore structures, bridges, engine components and load bearing parts of automobiles, ect. in order to ensure the long-term safety of the actual structures and to reduce cost together with the environmental load to the globe. It is a general view that in VHCF regime the crack

initiation is the life-controlling and consumes close to 99% of the total number of load cycles to failure. Hydrogen is one of the reasons for VHCF crack initiation in steels and it is trapped at internal inclusions, resulting in reduced the effective stress intensity threshold required for a crack to propagate.

Recently, hydrogen trapping using V-rich carbides was introduced to improve VHCF of a Cr–Mo low alloy steel by Weijun Hui of Beijing Jiaotong University and the co-authors, Yongjian Zhang and Xiaoli Zhao of Beijing Jiaotong University, Chao Zhou of Shenhua Guohua (Beijing) Electric Power Research Institute Co. Ltd., Kaizhong Wang and Wei Sun of Maanshan Iron & Steel Co. Ltd. and Han Dong of Central Iron and Steel Research Institute. Their work entitled “Very High Cycle Fatigue Properties of Cr–Mo Low Alloy Steel Containing V-rich MC Type Carbides” was published on Materials Science & Engineering A, Vol. 651, 2016, pages 311–320.

In this study, a commercial Cr–Mo low alloy steel microalloyed with V and Nb was used and its chemical

composition is shown in Table 2. Specimens for fatigue tests, hydrogen charging and tensile tests were machined from the as-hot rolled of the steel in the longitudinal direction. All the specimens were austenitized at 940°C for 45min, oil quenched and reheated to different tempering temperatures of 500°C and 600°C for 90min followed by air cooling. After the quenching and tempering (QT) treatment, part of the specimens were electrochemical hydrogen charged (CH). Some of the hydrogen-charged specimens were also exposed in air at room temperature for 24–360 hours to investigate hydrogen desorption behavior. Fatigue tests were conducted up to 10^9 cycles using an ultrasonic fatigue testing machine.

Table 2 Chemical composition of the steels (wt.%)

| C | Si | Mn | S | P | Cr | Mo | V | Nb | Al | N |
|------|------|------|-------|-------|------|------|------|------|------|-------|
| 0.43 | 0.10 | 0.35 | 0.015 | 0.005 | 0.99 | 0.37 | 0.27 | 0.03 | 0.01 | 0.008 |

The tempered specimens have mainly tempered lath martensitic microstructure with cementite particles within laths and on lath boundaries. Both TEM and physical–chemical phase analyses showed that only coarse undissolved MC and M₃C carbides were observed in the 500°C tempered specimen, whereas considerable amount of fine (length of ~10 nm and thickness of <1–2 nm) MC particles precipitated in the 600°C tempered specimen in addition to coarse undissolved MC and M₃C particles. The MC particles were identified as V-rich MC carbides by Energy-dispersive X-ray analyser (EDX) and physical chemical phase analyses. It is suggested that the fine V-rich particles precipitated during tempering at 600°C and those fine particles provided secondary hardening and hydrogen trapping, which influence the hydrogen absorption and desorption behavior and thus fatigue properties of the tested steel.

The introduced hydrogen content after the hydrogen charging is 4.22 ppm in the 600°C tempered specimen and 1.24 ppm in the 500°C tempered specimen. The higher hydrogen content in the 600°C tempered specimen is mainly owing to the precipitation of fine V-rich carbides at 600°C, which provide trapping sites for hydrogen. It is also revealed that most of the charged hydrogen is trapped at fine V-rich carbide interfaces rather than at inclusion interfaces in the 600°C tempered specimen.

The tensile strength and Vickers hardness are almost equal for the 600°C and 500°C tempered specimens under both the uncharged and hydrogen-charged conditions, as shown in figure 3 due to the secondary hardening occurred during 600°C tempering. Both fatigue strength and fatigue strength ratio of the 600°C tempered specimen are superior than that of the 500°C tempered specimen for both the uncharged and hydrogen-charged conditions (figure 3) though the 600°C

tempered + hydrogen-charged specimen possessed higher hydrogen content. No notable difference in fracture surfaces was observed between the hydrogen-charged and uncharged specimens. The authors indicated that compared to other high strength steels that they investigated previously, the influence of hydrogen on fatigue properties in the VHCF regime of the tested steel is comparatively small. It is suggested that improvement in fatigue properties in the VHCF regime of high strength steels could be obtained by the application of fine V-rich MC carbides owing to their hydrogen trapping effect.

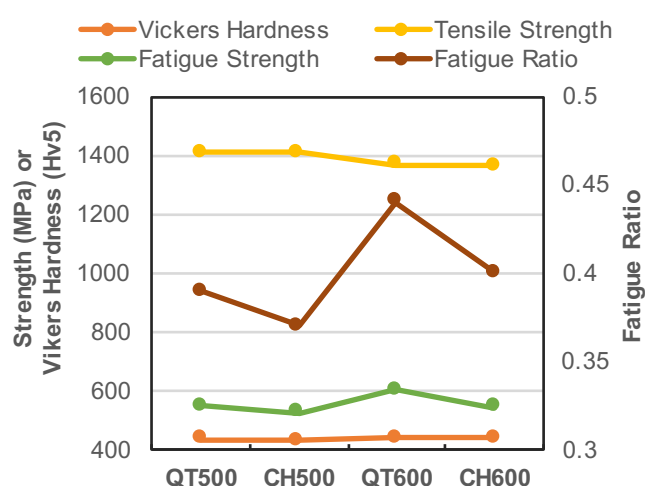


Fig. 3 Effect tempering temperature and hydrogen charge on the hardness, tensile and fatigue of the steel

Fine Vanadium Carbides Suppress the Hydrogen Embrittlement by Providing Hydrogen Trapping Sites in Tempered Martensitic Steel

Currently, there is an increasing demand for higher strength steels. However, with the use of steels of ultra-high strength level, hydrogen embrittlement (HE) becomes a serious issue. In steel, hydrogen can derive from production process, product assembling and finishing, or from service environment exposure. If hydrogen content reaches the critical value inside the steel, it can cause a loss in ductility or load carrying ability or cracking or catastrophic brittle failures at applied stresses well below the yield strength or even the normal design strength for the steels. This phenomenon often occurs in steels that show no significant loss in ductility, when measured by conventional tensile strengths, and is frequently referred to as hydrogen induced delayed brittle failure, hydrogen stress cracking or hydrogen embrittlement. The hydrogen embrittlement mechanism is governed by the hydrogen diffusion within the steels and the critical value of allowable diffusible hydrogen content decreases as the strength level increases.

The introduction of hydrogen trapping is a significant way to reduce the sensitivity of steel to hydrogen embrittlement. Vanadium based carbides are known to suppress the hydrogen embrittlement phenomenon by providing hydrogen trapping sites in steels. It is also reported that the de-trapping of hydrogen is very slow,

while the trapping presumably proceeds rapidly for steels containing VC precipitates.

A recent paper entitled “Effects of Vanadium Carbides on Hydrogen Embrittlement of Tempered Martensitic Steel” by Junmo Lee of Pohang University of Science and Technology, Republic of Korea and his co-workers was published in *Metals and Materials International*, Vol. 22, No. 3, 2016, pages 364-372. Four carefully designed V-free and V-added steels were used in this study and the chemical composition is given in Table 3. The steels were hot-rolled to a thickness of 13 mm. After rolling, the steels were austenitized for 30 min, and then quenched to 60°C in an oil solution. The steels were tempered at 500°C (V-free steel) or 570°C (V-added steels) for 60 min and then quenched in water. The different tempering temperatures were selected to maintain the strength of the investigated steels (~1.6 GPa in ultimate tensile stress (UTS)) for precluding a significant influence of mechanical strength on hydrogen embrittlement resistance. Hydrogen was charged into the specimens in a solution of 0.4% NaOH or a solution of 3% NH₄SCN and 0.3% NaCl at a charging current of 0.1-30 A·m⁻² for 48 h. A thermal desorption analysis (TDA) was used to measure the amount of hydrogen in the steels. Hydrogen embrittlement sensitivity was evaluated by using through a slow-strain-rate test (SSRT) to compare the fracture stress of uncharged and charged specimens.

Table 3 Chemical composition of the steels (wt.%)

| Steel | C | Si | Mn | S | P | Cr | V |
|--------|------|------|------|---------|---------|------|------|
| V-free | 0.60 | 2.01 | 0.20 | < 0.003 | < 0.003 | 1.01 | - |
| 0.2V | 0.60 | 2.05 | 0.20 | < 0.003 | < 0.003 | 0.99 | 0.20 |
| 0.5V | 0.60 | 2.03 | 0.20 | < 0.003 | < 0.003 | 1.00 | 0.50 |
| 1.0V | 0.61 | 2.03 | 0.20 | < 0.003 | < 0.003 | 0.99 | 1.01 |

All of the steels showed tempered martensitic microstructure with similar dislocation densities. Two types of V carbides: those that did not dissolve during the austenitizing step (undissolved carbides) and those that precipitated during the tempering step (precipitated carbides) were observed in the V-added steels. The undissolved carbides have a spheroidal shape with average particle size of 40-100 nm and the precipitated V carbides have a very thin plate shape with a thickness of 1-2 nm and a length of 10-30 nm. It is noted that the size and the number of undissolved V carbides increased as V content increased. It is of particular note that 0.2V steel had significantly fewer undissolved carbides compared to the other V-added steels. However, it was considered that the V-added steels contained similar amounts of precipitated carbides when tempered at the same temperature and time.

Thermal desorption analysis indicated that the V-added steels trapped more hydrogen than the V-free steel and hydrogen trapping showed a clear tendency to increase as V content increased. However, the SSRT results (figure 4) showed that hydrogen embrittlement resistance varied as 0.2V>0.5V≈1.0V>V-free steel. This tendency in hydrogen embrittlement resistance is different from the trend in hydrogen-trapping capability shown in the thermal desorption analysis results. The 0.2V steel exhibited the best HE resistance in terms of fracture stress after hydrogen charging. Excessive V contents in the 0.5V and 1.0V steels led to the formation of large undissolved carbides, which were less able to trap hydrogen than precipitated V carbides and gave rise to brittle fracture and decreased hydrogen embrittlement resistance.

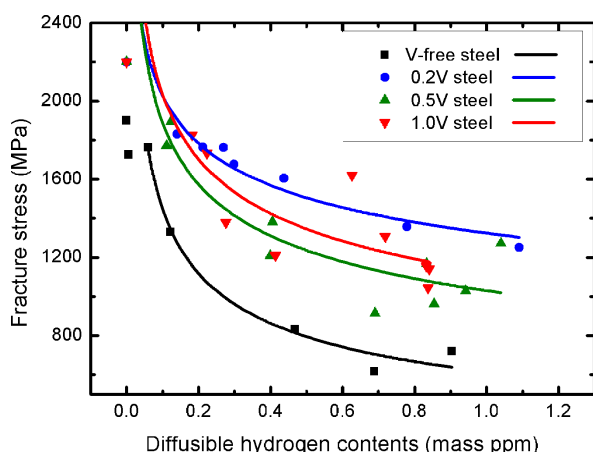


Fig. 4 SSRT Results showing the HE resistance of the steels

Optimisation of Cooling Process of V-N Microalloyed Steel Plate

Jing Zhang, et al. of University of Science and Technology Beijing has recently published a paper entitled “Microstructure, Precipitation, and Mechanical Properties of V-N-Alloyed Steel After Different Cooling Processes” in Metallurgical and Materials Transactions A, Vol. 47, No. 12, 2016, pages 6621-6631 to demonstrate their work on optimizing controlled cooling process in order to refine the microstructure and promote the precipitation strengthening of V(C,N), consequently increasing yield strength without loss of ductility and toughness of 600 MPa grade V-N-alloyed steel plate for architectural construction.

The experimental steel was microalloyed with V-N and the steel composition is shown in Table 4. The experimental steel was produced by intermediate frequency vacuum induction melting, and cast to a 39 kg ingot. The ingot was hot forged into slabs with a dimension of 50mmx50 mmx150mm at temperature range from 950°C to 1150°C and then air cooled to room temperature. The rolling schedule is illustrated in figure

The activation energy for hydrogen detrapping was calculated to be 14.1 kJ·mol⁻¹ for V-free steel and 27.4 kJ·mol⁻¹ for the 0.2V steel. The V-free steel desorbed hydrogen much faster than the V-added steel and it was revealed that most hydrogen did not diffuse out from V carbides even 24 h after the charging. Therefore, V carbides are considered to be non-diffusible hydrogen-trapping sites in this study.

This study suggests that improved hydrogen embrittlement resistance can be achieved by minimizing the size and amount of undissolved V carbides. To minimize the fraction of undissolved V carbides and to fully utilize the precipitated V carbides, V composition is recommended to follow the V solubility at a target austenitizing temperature.

5. Three cooling processes including direct air cooling, water cooling (15°C/s) to 750°C or 600°C followed by air cooling after hot rolling were used to study the effect of cooling on microstructure, V(C,N) precipitation and mechanical properties.

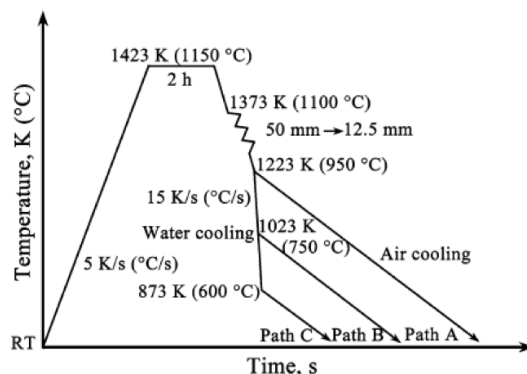


Fig. 5 Schematic diagram of the rolling schedule with three different cooling processes

Table 4 Chemical composition of the steels (wt. %)-1

| C | Si | Mn | S | P | V | N |
|------|------|------|-------|-------|------|-------|
| 0.23 | 0.75 | 1.55 | 0.004 | 0.007 | 0.23 | 0.019 |

Under three different cooling conditions, all microstructures are composed of polygonal ferrite and pearlite. Compared to the microstructure obtained from traditional direct air cooling (cooling path A), the grain size of ferrite is refined from 6.5± 0.49 μm to 4.6± 0.22 μm and the interlamellar spacing of pearlite decreases from 136± 8.8 nm to 45± 3.6 nm (figure 6) by the application of accelerated cooling path C. EBSD analysis revealed that the microstructural homogeneity for the cooling path C is higher than that for the other two cooling paths A and B. There is also a higher fraction of high misorientation angle grain boundaries in the hot-rolled steel followed by the cooling path C.

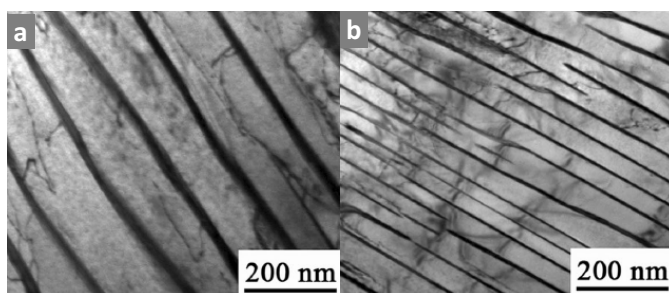


Fig. 6 TEM micrographs showing lamellar structure of pearlite for the experimental steel with (a) cooling path A and (b) cooling path C.

The TEM observation (figure 7) showed that the sheet spacing of the interphase V(C,N) precipitates is 23 to 26 nm for the cooling path A and about 21 nm for the cooling path B. The finest sheet spacing (14 to 17 nm) is obtained from the cooling path C. The volume fraction of fine (2 to 5 nm) random precipitates is also higher for the cooling path C than the cooling paths A and B.

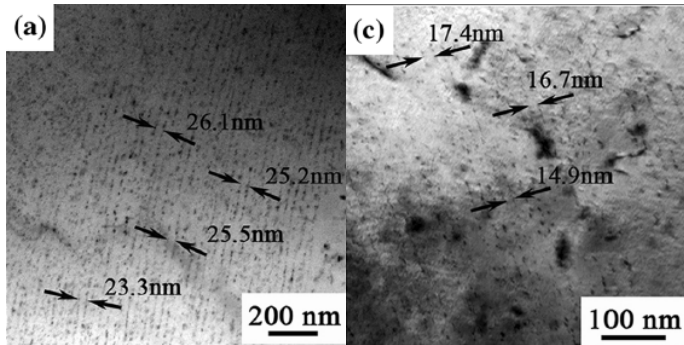


Fig. 7 TEM micrographs showing nano-scale interphase precipitates for the cooling path A (a) and the cooling path C (c).

Compared with the directly air-cooled steel, the application of accelerated cooling and lower finish cooling temperature significantly increases yield and tensile strength of the V-N microalloyed steel. Meanwhile, the total elongation and uniform elongation are almost similar for the three cooling processes and impact energy slightly increases with the cooling path C. The best combination of strength, ductility and toughness of the V-N microalloyed steel is obtained from cooling path C with 753 MPa yield strength, 922 MPa tensile strength, 22% total elongation, 11% uniform elongation and 36J impact energy at room temperature. The optimal mechanical properties obtained from the cooling path C are mainly attributed to the decrease of ferrite grain size, interlamellar spacing of pearlite, particle size and sheet spacing of nano-scale interphase precipitation of V(C,N), as well as the increase of volume fraction of fine random precipitation of V(C,N) with accelerated cooling rate and lower finish temperature.

Recent Vanadium Related Publication List

- A Comparison between Ultra-high-strength and Conventional High-strength Fastener Steels: Mechanical Properties at Elevated Temperature and Microstructural Mechanisms**, Carin Emmy Ingrid Christersdotter Ohlund, Mladena Lukovic, Jonathan Weidow, Mattias Thuvander and Sven Erik Offerman, ISIJ International, Vol. 56, No. 10, 2016, pp. 1874-1883.
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- Effects of Vanadium on the Continuous Cooling Transformation of 0.7 %C Steel for Railway Wheels**, Defect & Diffusion Forum, Vol. 367, 2016, pp. 60-67.
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Calendar of Technical Conferences and Seminars

| Date & Place | Steel Related Events |
|--|---|
| 22-23 February 2017 Jamshespur, India. | International Conference on Developments in Advanced High Strength Steel, Processing and Application (AHSS 2017) |
| 26 February-2 March 2017 San Diego, USA. | 2017 TMS Annual Meeting & Exhibition |
| 20-22 March 2017 Colorado School of Mines, USA. | ASPPRC Spring Sponsor Review Meeting |
| 08-11 May 2017 Tennessee, Nashville, USA. | The Iron & Steel Technology Conference and Exposition (AISTech 2017) |
| 24-26 May 2017 Brno, Czech Republic. | METAL 2017 - 26th International Conference on Metallurgy and Materials |
| 30 May-2 June 2017 Keystone, USA. | International Symposium on New Developments in Advanced High-Strength Sheet Steels |
| 4-7 June 2017 Atlanta, GA, USA. | Hot Sheet Metal Forming of High-Performance Steel (CHS2 2017) |
| 18-22 June 2017 Amsterdam, Netherland. | 5th International Conference on Steels in Cars and Trucks (SCT2017) |
| 16-18 August 2017 Qingdao, China. | The 7th International Conference on Modelling and Simulation of Metallurgical Processes in Steelmaking (STEELSIM2017) |
| 11-14 September 2017 Congress Graz, Austria. | 20th International Forgemastrs Meeting – IFM 2017 |
| 8-12 October 2017 Pittsburgh, USA. | Materials Science & Technology 2017 (MS&T17) |
| 12-16 November 2017 Tokyo, Japan. | 11th International Conference on Zinc and Zinc Alloy Coated Steel Sheet (GALVATECH) |
| Date & Place | Energy Storage Related Events |
| 14-16 February 2017 San Francisco, USA | 7th Annual Next-Generation Energy Storage |
| 28 February- 1 March 2017 London, UK | Energy Storage Summit |
| 9-10 March 2017 Miami, USA | 19th International Conference on Lithium Batteries |
| 20-23 March 2017 Fort Lauderdale, USA | International Battery Seminar & Exhibit |
| 8-12 May Berlin, Germany | 10th Energy Storage World Forum |
| 18-20 May 2017 Shenzhen, China | China International Battery Fair |
| 30 May- 2 June 2017 Munich, Germany | ees Europe |
| 19-22 June 2017 San Francisco, USA | 17th Annual Advanced Automotive Battery Conference (AABC) |
| 27-29 June 2017 Manchester, England | 8th International Flow Battery Forum (IFBF2017) |

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