

Issue No. 7 - May 2019

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Vanitec Limited

Hildenbrook House
The Slade
Tonbridge
Kent
TN9 1HR
UK

Tel: +44 (0)1732 240121
E-mail: info@vanitec.org
website: www.vanitec.org

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Recent Vanadium Related Publications

Vanadium Microalloying Provides Promising Comprehensive Mechanical Properties for Novel Ultra-high Strength Press Hardening Steels

Ultra-high strength press hardening steel (PHS) has been increasingly used to produce structural automobile components in order to meet specific crash-performance criteria and reducing vehicle weight. In the traditional press hardening process, the as-delivered strip of boron steel blank is heated in a furnace to its austenitisation temperature of around 900°C, formed in an internally cooled die set, and quenched under pressure to form a martensitic microstructure in the part, imparting high strength up to 1500 MPa. The press hardened components have excellent shape accuracy, but without the problem of springback. Currently, there is a demand for novel PHS providing ultra-high tensile strength of ~2000 MPa and superior crash ductility to further increase the crash safety and reduce vehicle weight. It has shown [1,2] that vanadium microalloying in traditional PHSs increases the strength and the ductility, and improves the resistance to hydrogen embrittlement. A recent study (Microstructure-Property Relationships of Novel Ultra-High-Strength Press Hardening Steels, Henri Järvinen, Mari Honkanen, Olli Oja, Martti Järvenpää, and Pasi Peura, Metallurgical and Materials Transactions A, vol. 50, no. 2, 2019, pp. 816-836.) demonstrated that vanadium microalloying, when combined with either Cr or Mo, provides a promising combination of mechanical properties of novel ultra-high strength press hardening steels.

Six steels including five laboratory manufactured 34MnB5-based steels (4 boron-alloyed (Steels A-D) + 1 non-boron-alloyed (Steel E)) and a commercial cold rolled and annealed 34MnB5 grade (1.1 mm in thickness) as reference steel (Ref.) were used to study the role of Ti and V when combined with Cr or Mo on the microstructure and property of the PH steels. The chemical compositions of the steels are shown in Table 1.

Steels A & B were Cr-alloyed and Steels C through E were Mo-alloyed. Steel A was microalloyed with a hyperstoichiometric amount of Ti (0.09 wt.%) in order to form Ti-based microalloy precipitates. Steels B through D were alloyed with a conventional Al-Ti-B concept but using a slightly reduced amount of Ti (~0.020

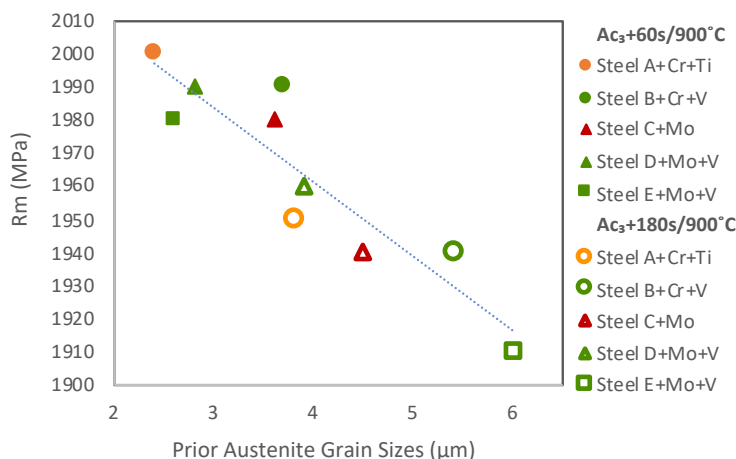


Fig. 1 Relationship between PAGS and tensile strength of the PH steels

Table 1 Chemical composition of the steels (wt%)

Steel	C	Si	Mn	Cr	Mo	V	Ti	Al	B	N	Ti/N
Ref	0.35	0.26	1.30	0.15	0.01	0	0.029	0.033	0.0026	0.0033	8.8
A+(Cr+Ti)	0.35	0.30	1.37	0.30	0	0	0.090	0.023	0.0022	0.0029	31.0
B+(Cr+V)	0.34	0.30	1.37	0.30	0	0.144	0.020	0.024	0.0019	0.0036	5.6
C+(Mo)	0.34	0.29	1.38	0.02	0.29	0	0.019	0.021	0.0019	0.0017	11.2
D+(Mo+V)	0.34	0.30	1.36	0.02	0.30	0.146	0.020	0.028	0.0017	0.0033	6.1
E+(Mo+V)	0.34	0.29	1.28	0.02	0.30	0.147	0.001	0.004	0.0001	0.0060	0.2

wt.%). All N in steels B through D should be combined with Ti to form TiN since their Ti/N ratio higher than the stoichiometric Ti/N ratio of 3.4. Steel E was deoxidized using the typical amounts of Si and Mn without normal Al addition and contained a slightly higher amount of N on purpose. Thus, the precipitation of TiN (and AlN) was thought to be eliminated in steel E, leaving a vast majority of N available for precipitation with V. Steels B, D, and E were microalloyed with V (~0.15 wt.%) to form V-based microalloy precipitates.

The laboratory steels were reheated at 1250°C for 60 minutes and hot rolled to 3-mm thickness using a laboratory rolling mill, and then cold rolled (CR) to 1.5-mm final thickness. The CR strip was cut into 100x60x1.5-mm sample sheets to be used in press hardening (PH) experiments, which were carried out by using custom-built PH equipment with a water-cooled flat die. Two austenitization cycles designated as Ac₃+60 s and Ac₃+180 s were carried out. After soaking, a sample was automatically transferred within 3 seconds to the die and subsequently quenched with a cooling rate of at least 80°C/s without deformation. After the PH experiments, another set of the die-quenched steel sheet samples were heated at 170°C for 20 min subjecting to the bake hardening (BH) heat treatment. Microstructure-property relationships were analysed after die quenching and the additional BH heat treatment using advanced methods of SEM and TEM microscopy, quasi-static tensile tests, and three-point bending tests.

The results showed that the microstructure of the PH steels was predominantly martensitic with very fine prior austenite grain sizes (PAGSs) (2-6 μm) after the die quenching. Fine precipitates were observed (1-10 nm) in the Ti microalloyed steel A and (1-20 nm) in the V microalloyed steels B, D and E in the PH condition after the austenitizing cycles. For the V containing steels, Steel E had the highest amount of fine precipitates after the short austenitizing cycle ($A_{c3}/60\text{s}$). The longer austenitization time ($A_{c3}/180\text{s}$) resulted in coarsening and decreased the number of fine precipitates. SEM analysis showed that the Ti microalloyed Steel A with a hyperstoichiometric amount of Ti contained the highest amount of coarse secondary phase particles/inclusions, whereas the measured amounts were the smallest in the V microalloyed Steel E. Both Ti and V microalloying results in prior austenite grain size (PAGS) refinement through the formation of stable nanosized precipitates and slightly increased strength. A correlation between PAGS and the strength of the PH steels was observed as shown in figure 1. In addition, there is also a correlation between precipitate size and PAGS, supporting that the presence of fine microalloy precipitates plays a significant role in the PAGS refinement.

The simulated BH treatment of 170°C/20 min showed significant effects on the mechanical properties of the PH steels by increasing the yield strength ($\Delta R_{p0.2}$) and post-uniform elongation but decreasing the tensile strength values due to tempering of martensite. The BH effects demonstrated dependency on the alloying concept since it is controlled by the PAGS as well as size and type of microalloy precipitates (figure 2). Finer PAGS and smaller precipitate size correlate clearly with larger $\Delta R_{p0.2}$.

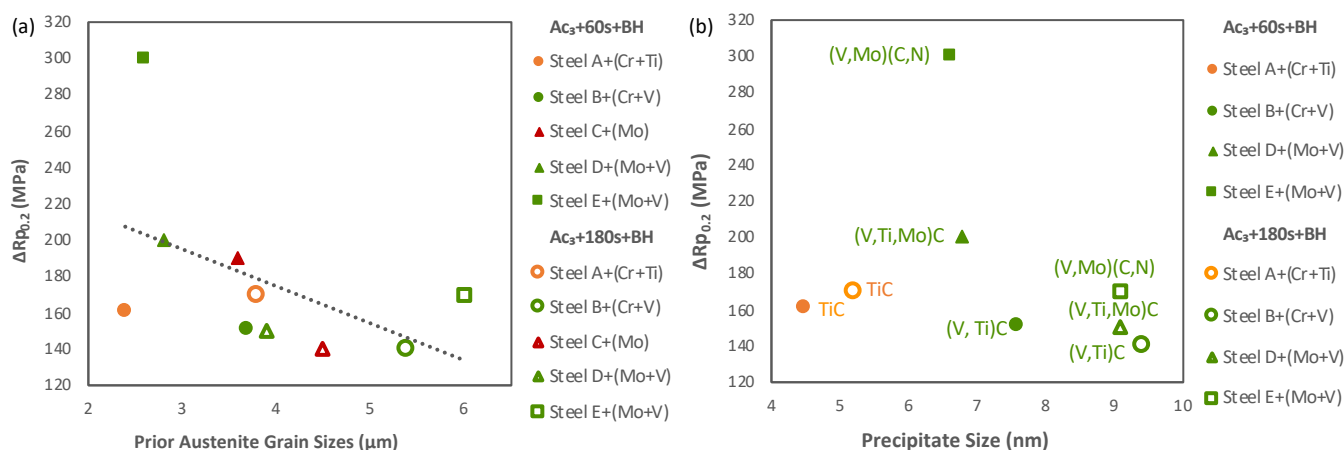


Fig. 2 Effect of bake hardening (BH) heat treatment on yield strength and increase in yield strength ($\Delta R_{p0.2}$) is related to PAGS (a) and precipitate size + type (b)

The V microalloyed Steel E with the alloying concept of 0.3Mo-0.15V-0.0060N (wt.%) exhibited a distinct BH behavior after the short austenitizing cycle. The $\Delta R_{p0.2}$ is 300 MPa for Steel E compared to 170 MPa on average for the other steels. In addition, the decrease in R_m is only 20 MPa for Steel E, but 130 MPa for other steels on average. It is suggested that the relatively large $\Delta R_{p0.2}$ value of Steel E may be attributed to the interactions between existing dislocations of martensite and very fine coherent (V,Mo)(C,N) precipitates, which compensate the tempering of martensitic matrix. The longer austenitization cycle decreased the $\Delta R_{p0.2}$ values of V-microalloyed steels, which may be connected with the loss in coherency.

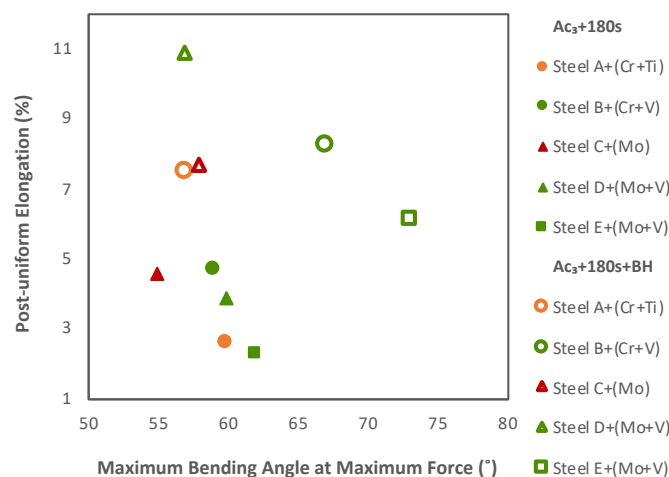


Fig. 3 Effect BH treatment on post-uniform elongation and bendability

The BH treatment also improved post-uniform elongation values for all the steels. However, for the measured maximum bending angle at maximum bending force (Three-point bending tests), significant improvements were observed only for the two V-microalloyed steels (Steels B and E) after the BH treatment as shown in figure 3. Steel E showed the best bendability after the BH treatment and the strongest positive effect of the BH treatment on the mechanical properties.

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Vanadium Improves the Resistance against Hydrogen Embrittlement

Currently, ultrahigh strength press hardening steel (PHS) with Al-Si coating experiences rapid growth in the anti-intrusion applications in the car body due to its improved safety and lightweight potential. However, hydrogen embrittlement remains to be a critical issue for PHS, which limits the industrial application of ultra-high strength aluminized PHS with tensile strength of > 1.5 GPa. PHS absorbs hydrogen during the austenitization in the hot press forming (HPF) process and this hydrogen uptake causes severe reduction of the plasticity of PHS. Aluminized PHS is considerably more sensitive to hydrogen uptake and the hydrogen induced loss of plasticity than uncoated PHS. A recently published work "Influence of Vanadium on the Hydrogen Embrittlement of Aluminized Ultrahigh Strength Press Hardening Steel, Lawrence Cho, Eun Jung Seo, Dimas H. Sulistiyono, Kyoung Rae Jo, Seong Woo Kim, Jin Keun Oh, Yeol Rae Cho, and Bruno C. De Cooman, Materials Science and Engineering, A 735, 2018, pp.448-455" demonstrated that vanadium additions, which serve to trap the hydrogen, considerably reduce the negative impact of the hydrogen uptake in aluminized 1800 MPa and 2000 MPa press hardening steels.

Two reference steels, 30MnB5 PHS grade (1800 MPa) and 35MnB5 PHS grade (2000 MPa) and two vanadium added steels (adding 0.2 wt% V to the reference steels), 0.2V-added 30MnB5 PHS and 0.2V-added 35MnB5 PHS, were used to evaluate the effect of vanadium additions on the resistance of the PHS to hydrogen embrittlement. All of the steels contain 10–20 ppm B for enhancing the hardenability and the formation of BN was avoided by the additions of 0.03 wt.% Ti. The industrially cold-rolled, full hard PHSs were continuously annealed in N₂ + 5 vol% H₂ gas atmosphere and hot dip aluminized at 680°C in an Al-10%Si bath. The continuously annealed steels were then austenitized by heating to an austenitizing temperature of 900°C and isothermal holding for 5 min in an electrically-heated furnace. After the austenitizing treatment, the steels were transferred to a laboratory hydraulic HPF simulator where the specimens were quenched between two flat water-cooled dies to room temperature. During the die quenching, the austenite was transformed to lath martensite. In this study, hydrogen was expected to naturally absorbed in the aluminized steel specimens during the austenitizing heat treatment as a result of high temperature atmospheric corrosion of the Al-Si coating due to the water vapor in the furnace atmosphere.

Microstructure of the PHSs was examined by field emission scanning electron microscopy (FE-SEM), electron backscattering diffraction (EBSD), and field emission transmission electron microscopy (Fe-TEM). The results showed that the vanadium addition to the PHSs results in the refinement of prior austenite grain size, packet size, and block size of the lath martensite, as shown in figure 4. The precipitation behavior of vanadium during the processing of the V-added PHSs was investigated by quantitative analysis using the electrochemical extraction of the precipitates. Most of the vanadium was found to remain in solid solution in the as-rolled condition and 20–30% of the added vanadium was precipitated as fine VC particles in the lath martensitic matrix in the PHSs in the press-hardened state. The grain refinement and precipitation strengthening of VC results in ~40 MPa increase in the strength of the PHS.

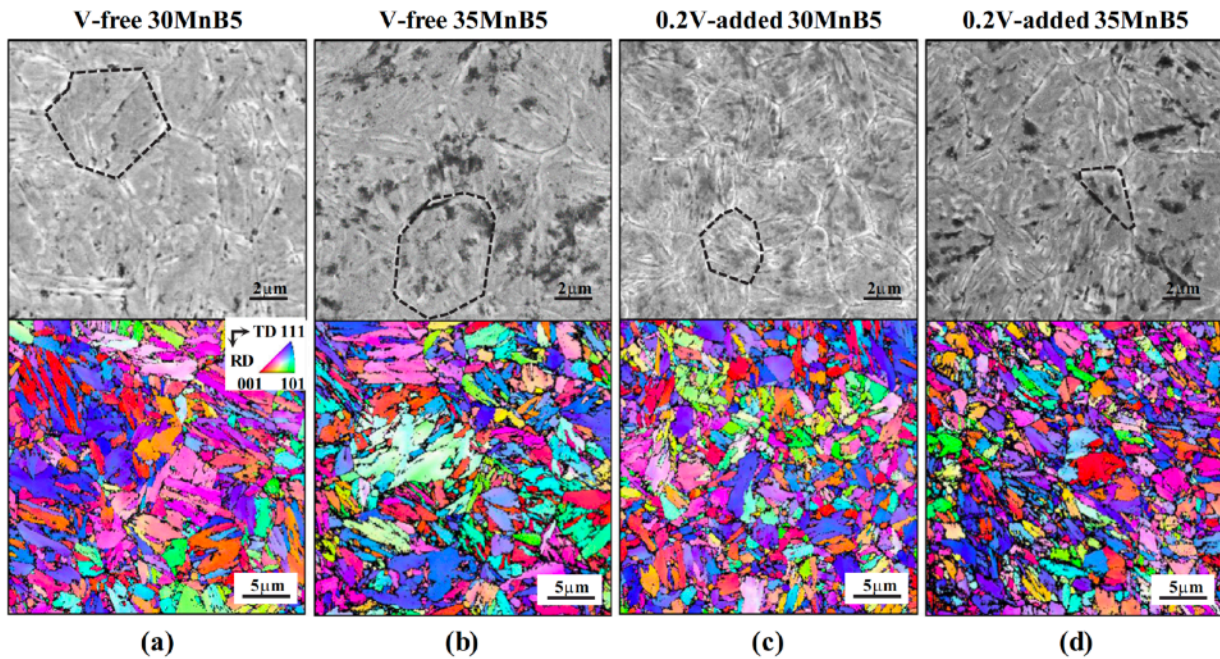


Fig. 4 SEM micrographs (top row) and EBSD inverse pole figure maps (bottom row) showing the fully martensitic microstructures of the aluminized PHSs and refined the martensitic microstructures in the V-added PHSs

The study demonstrated that diffusible hydrogen was introduced in the aluminized PHSs during the austenitization stage of the HPF process and the hydrogen uptake was accelerated due to the Al reduction of the water vapor in the furnace gas atmosphere during the austenitization. Hydrogen was trapped in the lath martensite and inhibited from escaping from the martensite matrix by the presence of the reacted alloy coating layer at the surface. The ductility of the aluminized V-free PHSs was clearly reduced due to the hydrogen embrittlement, which causes an early fracture of the 30MnB5 and 35MnB5 PHSs at an engineering strain of 4.1% and 2.3% respectively during the tensile test at room temperature, however the 0.2V-added PHSs exhibited the normal ductile fracture behaviour with a significant improvement of the ductility (total elongation \approx 5.5%), as shown in figure 5, indicating that the vanadium addition

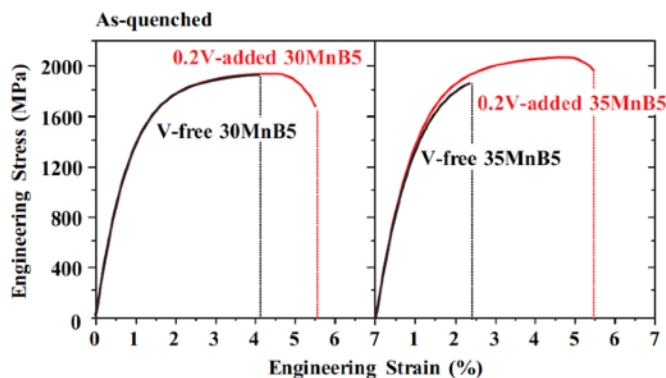


Fig. 5 Room temperature tensile properties of the aluminized V-free PHSs and the aluminized 0.2V-added PHSs in the as-quenched state, i.e. immediately after the HPF process.

improves the resistance against hydrogen embrittlement. In addition, the vanadium addition transforms the fracture mechanism from largely cleavage feature with intergranular cracks in the V-free PHSs to ductile with no intergranular cracks in the V-added PHSs. It is suggested that the key role of vanadium on the hydrogen embrittlement behavior of the PHSs is the enhancement of the hydrogen trapping capacity through precipitation of fine VC particles, thus significantly reducing the hydrogen diffusion coefficient. Furthermore, the refinement of martensite substructure in the V-added PHSs creates many more block and packet boundaries, leading to the depletion of the hydrogen content per unit boundary area. As a result, the vanadium addition effectively increases the critical diffusible hydrogen concentration below which the hydrogen embrittlement does not occur.

Remarkable Improvement of Creep Strength by Vanadium Addition in 2.25Cr Heat-Resistant Steel

Vanadium is a key element in heat resistant steels, where 0.2-0.3 wt.% V is added to provide good creep strength. Vanadium combine with C and/or N to precipitate as stable fine carbides, nitrides or carbonitrides, which enhance tensile strength at elevated temperature and creep rupture strength, and improve resistance to in-service degradation phenomena, such as temper embrittlement, high temperature hydrogen attack and hydrogen embrittlement. In order to meet the requirements of advanced boilers such as higher steam temperature and pressure, a research was carried out at Pohang University of Science and Technology, South Korea to design a low-Cr heat-resistant steel (T/P23) that has higher creep strength than conventional 9 wt.% Cr steels by increasing vanadium content greater than ASTM A213 standard range (0.20-0.30 wt.%V). The study (Remarkable Improvement of Creep Strength by Vanadium Addition in

2.25Cr Heat-Resistant Steel, Hyun Je SUNG and Sung-Joon KIM, ISIJ International, Vol. 58, No. 11, 2018, pp. 2142–2146) showed that increase of V content to >0.4 wt.% eliminates coarse $M_{23}C_6$ and allows only precipitation of MX carbonitrides in the T/P23 heat-resistant steel, resulting in remarkable improved creep strength, which is superior than that of T/P92 steel (9Cr-1.8W-0.5Mo-VNbTi).

Table 2 Chemical composition of the steels (wt%)

Steel	C	Si	Mn	P	S	Cr	Mo	V	Al	W	Nb	N	B	Ti
V255	0.100	0.318	0.509	0.021	<0.002	2.24	0.05	0.255	0.019	1.55	0.049	0.010	0.0013	0.016
V620	0.103	0.317	0.534	0.020	<0.002	2.24	0.03	0.620	0.023	1.51	0.049	0.010	0.0008	0.011
V798	0.098	0.318	0.512	0.019	<0.002	2.26	0.03	0.798	0.017	1.54	0.049	0.013	0.0009	0.010

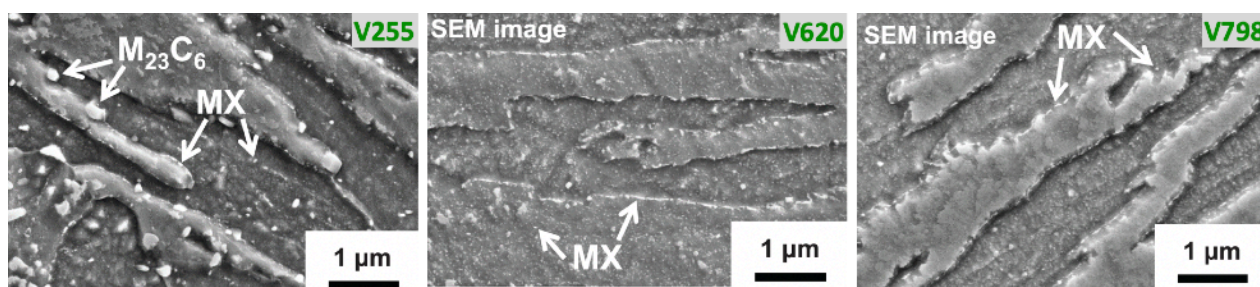


Fig. 6 SEM micrographs showing $M_{23}C_6$ carbides and MX carbonitrides in Steel V255, and only fine MX carbonitrides in Steels V620 and V797

Three T/P23 steels with vanadium content of 0.255 wt.%, 0.620 wt.% or 0.798 wt.% were used in the study and the chemical composition of the steels is shown in Table 2.

The ingots were homogenized at 1200°C for 1 h and hot-rolled to 12-mm thick plates. Rectangular bars machined from the plates in the hot-rolling direction were heat treated at 1050°C for 1 h followed by air cooling and subsequent tempered at 750°C for 0.5 h under Ar atmosphere. Cylindrical specimens machined from the bars were used to assess their creep strength at 600°C without soaking in ambient condition, using conventional creep testers (ATS 2320).

Microstructural examination using field-emission scanning electron microscopy (FE-SEM) and field-emission transmission electron microscopy (FE-TEM) showed that all specimens after tempering at 750°C for 0.5 h contained tempered bainitic microstructure and had similar narrow plate-like bainite sub-grains. Thermo-Calc calculation predicted that the Gibbs free energy difference for $M_{23}C_6$ carbide formation becomes positive when $[V] > 0.4$ wt.% at the tempering temperature of 750°C and the creep test temperature of 600°C, indicating that this change would discourage the formation of $M_{23}C_6$ carbide in Steels V620 and V798. Consistent with this prediction, it is confirmed by FE-SEM and FE-TEM that after the tempering, large Cr-rich $M_{23}C_6$ carbides (~250 nm) on boundaries, together with MX carbonitrides were observed in Steel V255, but only fine MX carbonitrides (< 20 nm) along boundaries and in the matrix were found in Steels V620 and V798 as shown in figure 6. The MX carbonitrides are mainly V-rich carbonitrides. The amount of V-rich MX carbonitrides at tempering temperature of 750°C and at the creep test temperature of 600°C increased significantly as vanadium content increased from 0.255 wt.% V to 0.620 wt.% V, but not with further increase to 0.798 wt.% V.

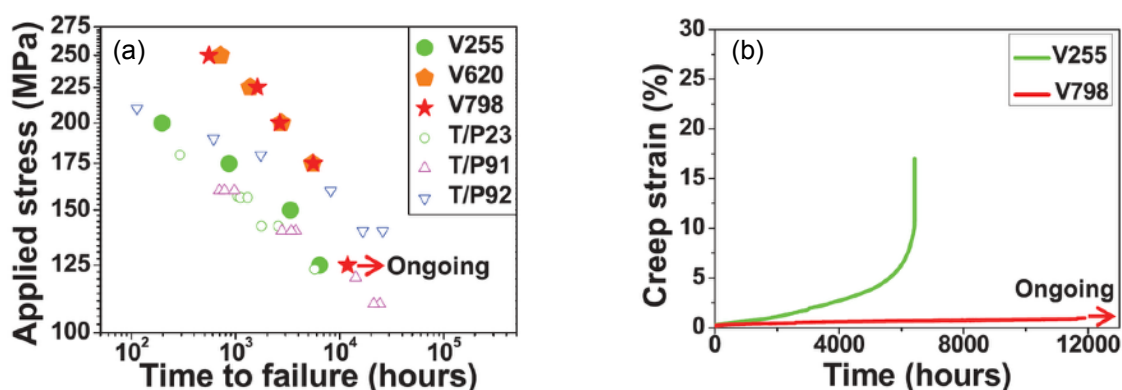


Fig. 7 Fig. 7 (a) Creep test results of V255, V620 and V798 together with ASTM T/P23, T/P91 and T/P92 steels performed by National Institute for Materials Science (NIMS), Japan, (b) measured creep strain under 125 MPa at 600°C.

Creep lives of Steel V255 were similar to those of T/P23 and T/P91 heat-resistant steels, examined by National Institute for Materials Science (NIMS), Japan [3]. The time to failure remarkably increased with increasing vanadium content from 0.255 wt.% to 0.620 wt.%, but Steel V620 and Steel V798 showed similar creep lives as shown in figure 7a. Both Steel V620 and Steel V798 are stronger than T/P92 steel (9Cr-1.8W-0.5Mo-VNbTi), based on NIMS data [3]. The V255 ruptured at 6427 h under 125 MPa at 600°C, whereas the V798 has endured applied stress for ~12000 h with very low creep deformation. This test is still ongoing (figure 7b).

It is suggested that the main reason for the improved creep strength of Steels V620 and V7698 is the increase of vanadium content greater than 0.4 wt.%, which leads to the elimination of coarse $M_{23}C_6$ and precipitation of only V-rich nano-sized carbonitrides in the T/P23 heat-resistant steels. The fine V-rich carbonitrides effectively stabilizes sub-boundaries of bainite microstructure, so coalescence of neighboring sub-boundaries and subsequent creep strength loss are suppressed. In addition, finely-dispersed V-rich carbonitrides also increases creep strength by providing precipitation hardening.

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Calendar of Technical & Trade Conferences

Date & Place	Vanitec Events
8 July 2019, Lyon, FRANCE	5th Vanitec ESC Meeting
8 & 9 October 2019, London, UK	97th Vanitec Meeting
7 & 8 April 2020, Las Vegas, USA	98th Vanitec Meeting

Date & Place	Steel Related Events
2019	
6-9 May 2019, Pittsburgh, USA	The Iron & Steel Technology Conference and Exposition (AISTech2019)
2-5 June 2019, Luleå, SWEDEN	The 7th International Conference on Hot Sheet Metal Forming of High-Performance Steel (CHS² 2019)
24-28 June 2019, Dusseldorf, GERMANY	4th European Steel Technology & Application Days (METEC & 4th ESTAD 2019)
18-22 August 2019, Xi'an, CHINA	The 10th Pacific Rim International Conference on Advanced Materials and Processing (PRICM-10)
30 September - 03 October 2019, Oregon, USA	The Materials Science & Technology Conference and Exhibition (MS&T19)
1-4 October 2019, São Paulo, BRAZIL	11th International Rolling Conference (IRC 2019)
2020	
4-7 May 2020, Ohio, USA	The Iron & Steel Technology Conference and Exposition (AISTech2020)
31 May-5 June 2020, Vienna, AUSTRIA	THERMEC'2020
14-18 June 2020, Milan, ITALY	6th Steels in Cars and Trucks (SCT) 2020
21-25 June 2020, Vienna, AUSTRIA	Galvatech 2020
24-26 August 2020, Shenyang, CHINA	6th International Conference on ThermoMechanical Processing (TMP2020)
9-11 September 2020, Sheffield, UK	Euro Steel 2020
16-18 November 2020, SOUTH KOREA	2nd Automotive Steel International Conference (ASIC2020)

Date & Place	Energy Storage Related Events
2019	
14-17 May 2019, Munich, GERMANY	ees Europe 2019
13-14 June 2019, Sydney, AUSTRALIA	Australian Energy Storage Conference and Exhibition 2019
9-11 July 2019, Lyon, FRANCE	International Flow Battery Forum
9-11 July 2019, San Francisco, USA	ees North America 2019
11-12 September 2019, Guangzhou, CHINA	Energy Storage China (ESC) 2019
10-12 October 2019, Shenzhen, CHINA	4th China Energy Storage Exhibition (CESE2019)
16-18 October 2019, Rome, ITALY	Energy Storage World Forum
5-7 November 2019, San Diego CA, USA	Energy Storage North America 2019
20-21 November 2019, Santa Clara, USA	Energy Storage Innovations 2019
2020	
January, 2020, New Delhi, INDIA	Energy Storage India (ESI) 2020
25-26 February 2020, London, UK	Energy Storage Summit 2020
10-12 March 2020, Dusseldorf, GERMANY	Energy Storage 2020
24-25 March 2020, Fort Lauderdale, USA	Energy Storage Americas 2020

Date & Place	Trade Events
25-27 September 2019, Chicago, USA	North American Ferroalloys 2019
20-22 October 2019, Miami, USA	CRU Ryan's Notes Ferroalloys USA 2019
17-19 November 2019, Budapest, HUNGARY	International Ferroalloys Conference 2019
22-23 May 2019, Amsterdam, NETHERLANDS	CRU Ryan's Notes Ferroalloys Europe 2019

Vanitec Members

<u>AMG Vanadium, Inc.</u> 60790 Southgate Road Cambridge OH 43725 USA Tel: +1 740 435 4600 Email: vanadium@amg-v.com www.amg-v.com	<u>Atlantic Vanadium Pty Ltd.</u> Level 24, Allendale Square 77 St Georges Terrace Perth, WA, 6000 Australia Tel: +61 8 6141 7100 Email: info@atlantictld.com.au www.atlantictld.com.au	<u>Australian Vanadium</u> Level 1, 85 Havelock Street West Perth WA 6005 Australia Tel: +61 8 9321 5594 Email: info@australianvanadium.com.au www.australianvanadium.com.au
<u>Bear Metallurgical Company</u> 679 East Butler Road Butler, PA 16002-9127 USA Tel: +1 724 431 2800 Email: info@bearmet.com www.bearmet.com	<u>BlackRock Metals</u> 1080 Beaver Hall Hill Suite 1606 Montreal Quebec H2Z 1S8 Canada Tel: +1 514 316-7281 www.blackrockmetals.com	<u>Bushveld Vametco</u> Main Mothotlung Road 0250 Brits North West South Africa Tel: +27 21 318320 Email: info@bushveldminerals.com www.bushveldminerals.com
<u>HBIS Group Chengsteel Company</u> Luanhe Town Shuangluan District Chengde Hebei 067002 China Tel: +86 314 4378909 Email: cg@cdsteel.cn www.cdsteel.com	<u>Chengde Jianlong Special Steel</u> Xinglong County Chengde City Hebei, 067201 China Tel: +86 314 5316566 Email: bfr00@163.com www.ejianlong.com	<u>China Iron & Steel Research Institute Group</u> No.76 Xueyuan Nanlu Haidian, Beijing 100081 China Tel: +86 10 62182761 Email: chenxuehui@cisri.com.cn www.cisri.com
<u>De Sheng Gruop</u> No. 8, Tonghe Road Shawan District Leshan, Sichuan China Tel: +86 0833 3480171 Email: 247222584@qq.com www.scdesheng.com	<u>Duferco S. A.</u> Via Bagutti, 9 6900 Lugano Switzerland Tel: + 41 91 822 56 00 Email: info@duferco.com www.duferco.com	<u>Energy Storage Solutions</u> Avenida de Barajas 32 Parque Empresarial Omega Edificio A, 28108 Alcobendas Spain www.energystoragesolutions.com
<u>Evraz NTMK</u> 1, Metallurgov Street Nizhniy Tagil Sverdlovskaya oblast 622025 Russia Tel: +7 3435 497270 Email: post@ntmk.ru www.ntmk.ru	<u>Evraz Vanady Tula</u> 1, Przhevalskogo Street Tula, 300016 Russia Tel: +7 4872 466 900 Email: info@vanady.com http://vt.evraz.com	<u>Ferrovan Oy</u> Asemakatu 37 F1-90100 Oulu Finland Tel: +358 8 370 090 Email: info@ferrovan.com www.ferrovan.com
<u>Glencore plc</u> P. O. Box 3620 Brits, 0230 South Africa Tel: +27 12 3180700 www.glencore.com	<u>Largo Resources Ltd.</u> 55 University Ave. Suite 1101, Toronto Ontario, M5J 2H7 Canada Tel: +1 416 861 9797 Email: info@largoresources.com www.largoresources.com	<u>New Zealand Steel Ltd.</u> Mission Bush Road Private Bag 92121 Auckland 1142 New Zealand Tel: +64 9 375 8999 www.nzsteel.co.nz

<u>Oxkem</u> 117 Loverock Road Reading RG30 1DZ UK Tel: +44 1189 522929 Email: info@oxkem.com www.oxkem.com	<u>Panzhihua Iron & Steel Group</u> Xiangyang Village East District Panzhihua, Sichuan 617067 China Tel: +86 28 87546705 Email: qinmingvip@vip.163.com www.pzhsteel.com.cn	<u>QEM Limited</u> Level 6 50 Appel Street Surfers Paradise 4217 Australia Tel.: +61 7 3303 0161 www.qldem.com.au
<u>Riverside Specialty Chemicals Inc.</u> 6 West 79th Street Suite 7W New York NY 10024 USA Tel.: +1 49 9122 8887480 www.riverchem.com	<u>Technology Metals Australia Limited</u> Suite 9, 330 Churchill Avenue PO Box 866 Subiaco WA 6904 Australia Tel.: +61 8 6489 1600 www.tmtlimited.com.au	<u>Treibacher Industrie AG</u> Auer-von-Welsbach-Straße 1 9330 Althofen Austria Tel: +43 42 62 5050 Email: treibacher@treibacher.com www.treibacher.com
<u>U308 Corp</u> 401 Bay Street, Suite 2702 Toronto, ON M5H 2Y4 Canada Tel: +1 416 868 1491 www.u3o8corp.com	<u>VanadiumCorp Resource Inc.</u> Suite #400 1505 West 2nd Vancouver, BC, V6H 3Y4 Canada Tel: +1 604 385 4485 Email: sp@vanadiumcorp.com www.vanadiumcorp.com	<u>Vanchem Vanadium Products (Pty) Ltd.</u> Ferrobank Van Eck Drive, Witbank Emalahleni, 1034 South Africa Tel: +27 13 696 6001 www.vanchemvanadiumproducts.com
<u>Wogen Resources Ltd.</u> 4 The Sanctuary Westminster London SW1P 3JS UK Tel: +44 20 7222 2171 www.wogen.com		