FTP/4-5Ra

Optimisation of production method of a nanostructured ODS ferritic steels

P. Unifantowicz[1], J. Fikar[1], P. Spätig[1], C. Testani[2], F. Maday[2], N. Baluc[1], M.Q. Tran[1]

[1]Fusion Technology-Materials, CRPP EPFL, Association EURATOM-Confédération Suisse, 5232 Villigen PSI, Switzerland

[2]ENEA CR Cassaccia, 2400-00100 Rome, Italy

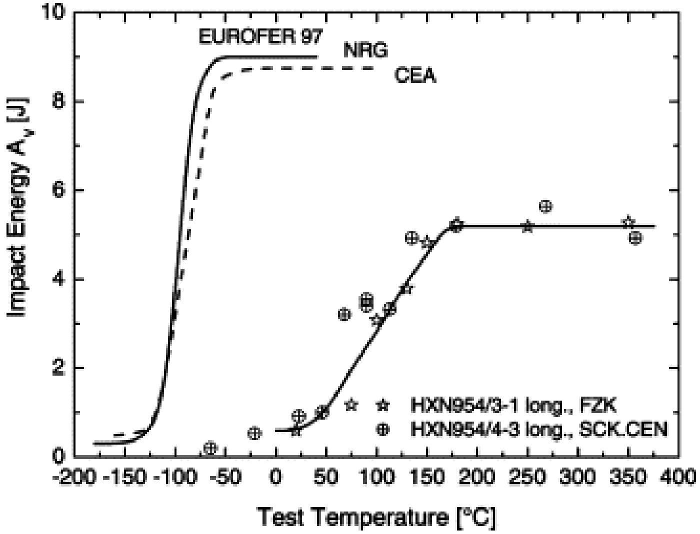
FTP/4-5Rb

Low Activation Vanadium Alloys for Fusion Power Reactors - the RF Results

V.M. Chernov, M.M. Potapenko, V.A. Drobyshev, D.A. Blokhin, N.I. Budylkin, E.G. Mironova, N.A. Degtyarev, I.N. Izmalkov, A.N. Tyumentsev, I.A. Ditenberg, K.V. Grinyaev, A.I. Blokhin, N.A. Demin, N.I. Loginov, V.A. Romanov, A.B. Sivak, P.A. Sivak, S.G. Psakhie, K.P.Zolnikov

Bochvar High Technology Research Institute of Inorganic Materials, Moscow, Russia
Tomsk State University, Tomsk, Russia
Leypunsky Insitiute of Physics and Power Engineering, Obninsk, Russia
NRC « Kurchatov Institute», Moscow, Russia
Institute of Physics Strength and Material Science, SB RAS, Tomsk, Russia

INTRODUCTION

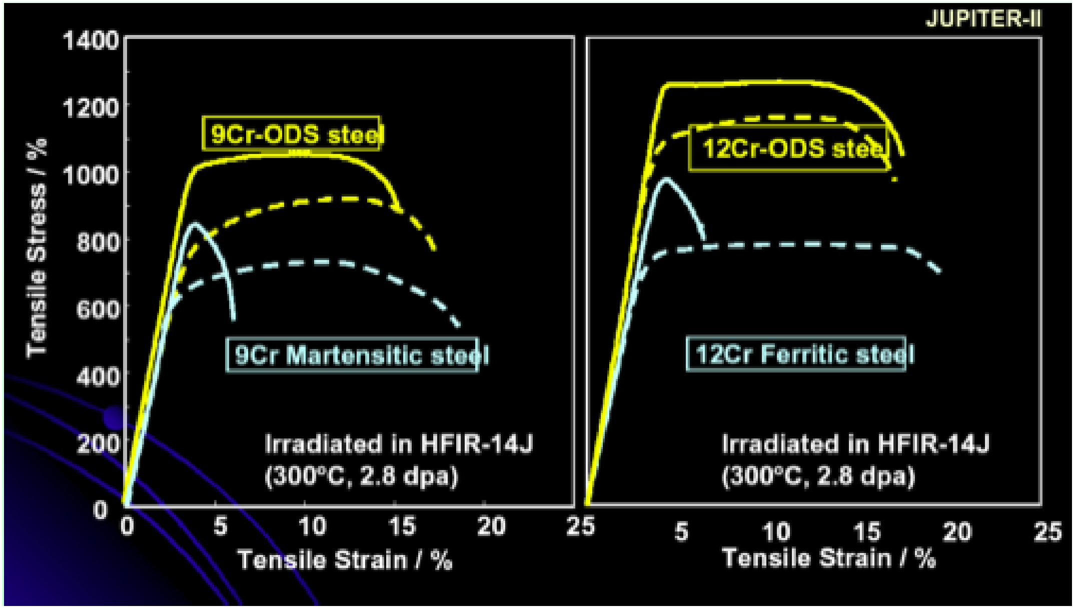


Test temperature dependence of total absorbed energy of ODS-EUROFER (0.3 wt% Y_2O_3) in comparison with RAFM steel EUROFER97

R. Lindau et al., JNM, 307–311, Part 1, 2002

Reduced Activation Ferritic ODS steels:

first choice candidates for fusion application



Neutron Irradiation
A. Kimura et al, ISFNT-7, May 2005

INTRODUCTION

Reduced Activation Ferritic ODS steels:

COMPOSITION

Iron matrix	
14% Cr	provides stability of ferritic structure, resistance to corrosion
W	improves thermal stability of the alloy
Ti, YO	nano-oxides improve resistance to creep, fatigue and radiation damage

PRODUCTION

- Powder metallurgy

  - Mechanical alloying

  - Powder compaction using hot extrusion (HE) or hot isostatic pressing (HIP)

- Thermo mechanical treatment

METHODOLOGY

- Powders mixed in Ar atmosphere
- Powder mixtures transferred in the attritor in a container filled with Ar
- Milling in attritor in controlled H_2 atmosphere for total 80h for elemental powders and 8h for mixture of pre-alloyed powder and reinforcement particles

Powder contamination:

Concentration of oxygen and nitrogen in the powders Criterion for selection of substrates and milling time.
E – elemental; P – pre-alloyed Fe14Cr2W0.3Ti base alloy.

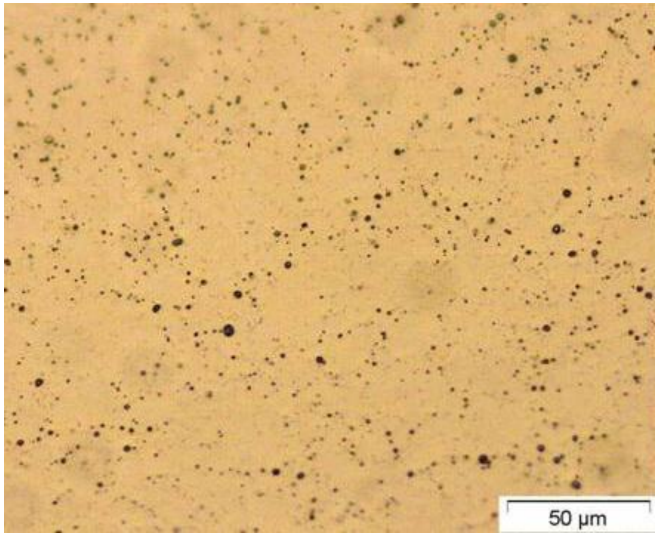
	As mixed $E+0.3Y_2O_3$	$E+0.3Y_2O_3$ 40h	$E+0.3Y_2O_3$ 80h	As-mixed $P+0.3Y_2O_3$	$P+0.3Y_2O_3$ 8h
wt. % O_2	0.44	0.53	0.65	0.15	0.27
wt. % N_2	0.04	0.06	0.06	0.01	0.06

Hot Cross Rolling:

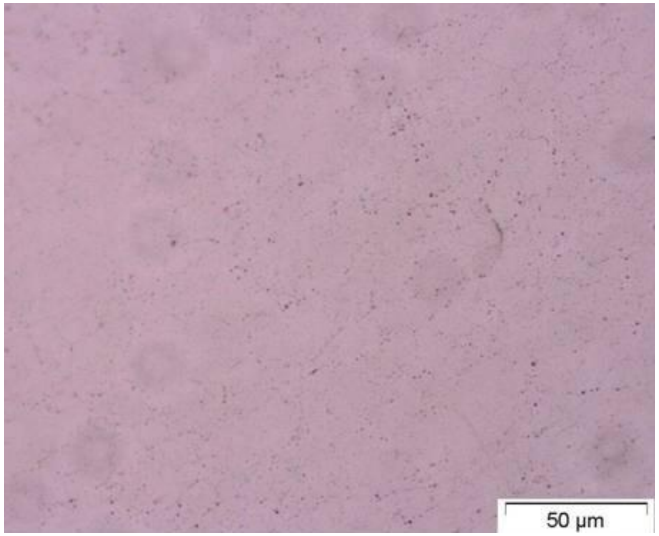
- Performed at the CSM Center (ENEA)
- Two directional rolling at 800C
- Highest degree of deformation 80% reduction of thickness (ROT)

MICROSTRUCTURE

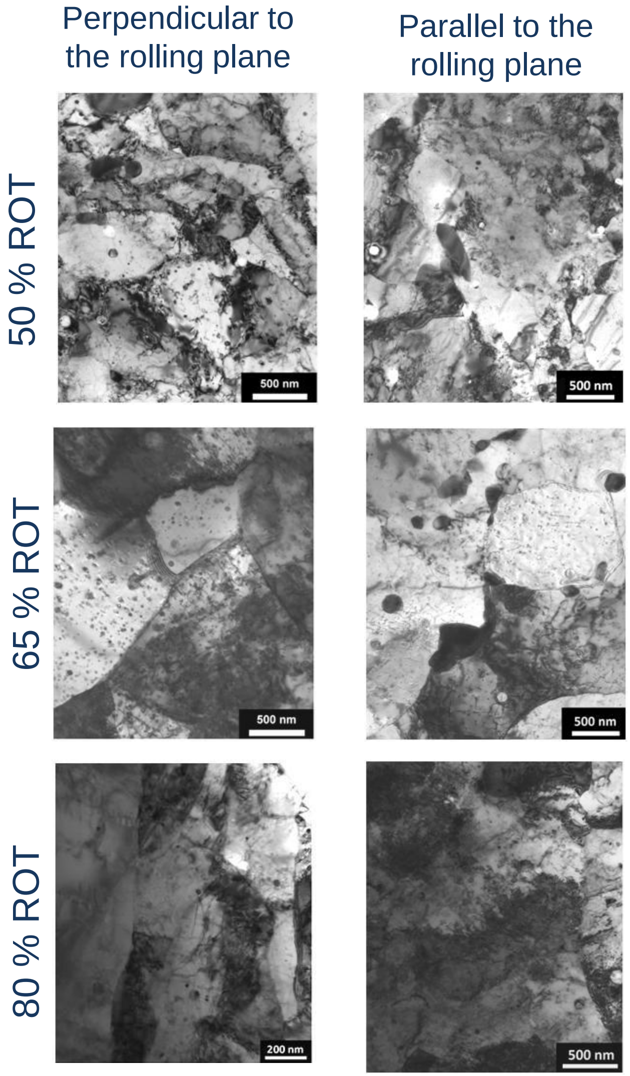
HIP: Large pores



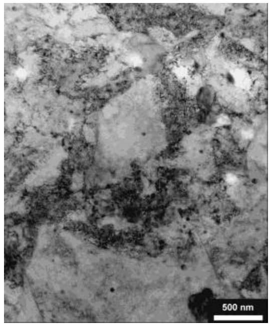
HCR: Finer structure



MICROSTRUCTURE



HIP



Average grain size:

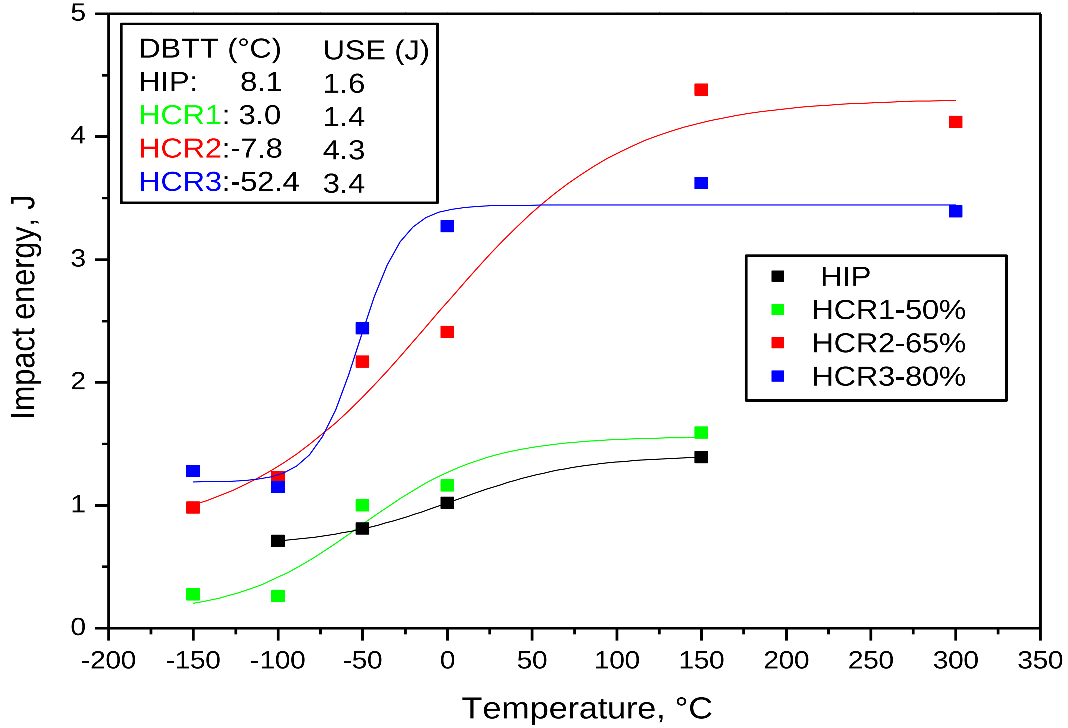
HIP: 0.3 µm
HCR 65% ROT: 0.5 µm

Average oxide diameter:

HIP: 6 nm
HCR (all ROT): 10 nm

EFFECT OF HOT-CROSS ROLLING

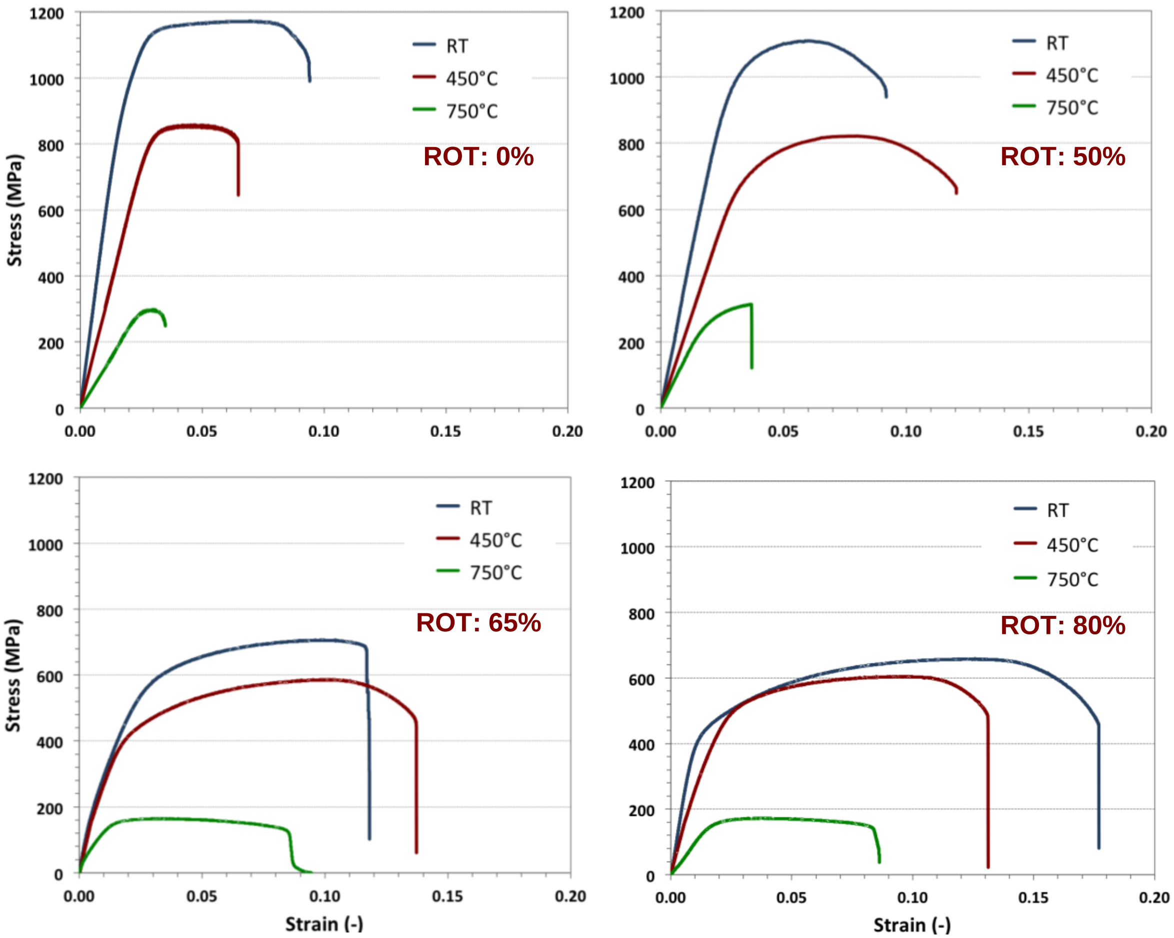
Charpy impact tests:



- Low upper shelf energy for HIP and 50% ROT HCR samples, i.e. low toughness in the plastic fracture regime
- Higher upper shelf energy and lower DBTT for 65 and 80% ROT (-50° C for 80% ROT)

EFFECT OF HOT-CROSS ROLLING

Tensile tests:



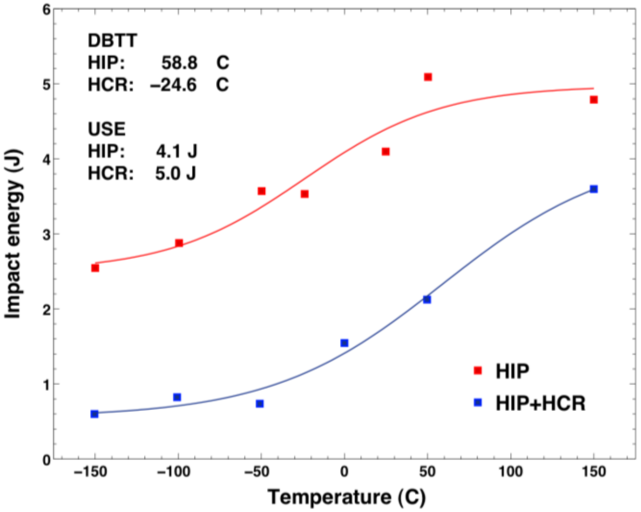
	ROT: 0%			ROT: 50%		
Test T (°C)	25	450	750	25	450	750
R_m (MPa)	1173	858	299	1109	821	313
$R_{p0.2}$ (MPa)	1053	801	281	937	673	250
ε	0.095	0.065	0.043	0.092	0.12	0.037
ε_u	0.02	0.028	0.024	0.027	0.033	0.019

	ROT: 65%			ROT: 80%		
Test T (°C)	25	450	750	25	450	750
R_m (MPa)	1173	858	299	1109	821	313
$R_{p0.2}$ (MPa)	1053	801	281	937	673	250
ε	0.095	0.065	0.043	0.092	0.12	0.037
ε_u	0.02	0.028	0.024	0.027	0.033	0.019

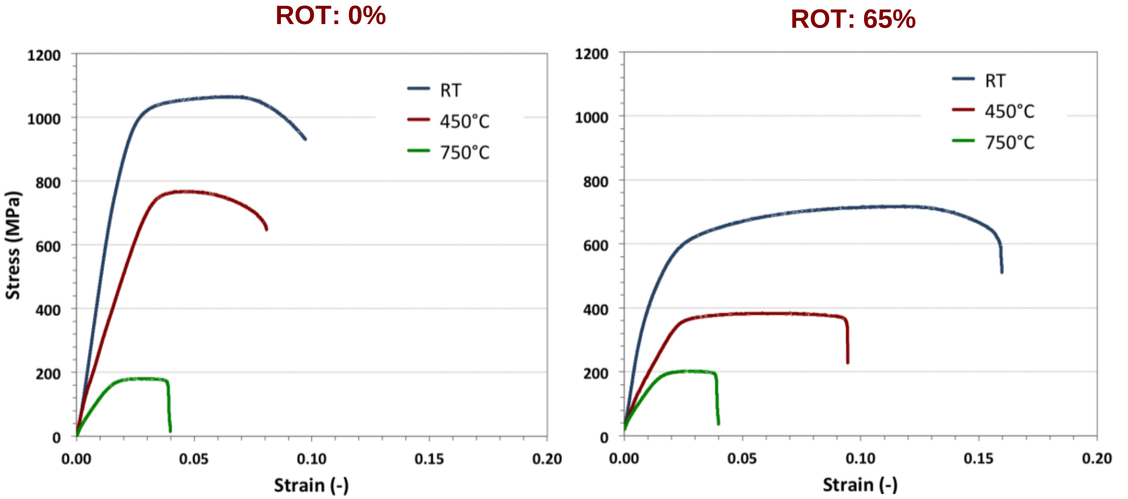
- Significant reduction of tensile strength in 50% and 65% ROT samples
- Smooth change of slope on the engineering strain-stress curves in 50% and 65% ROT compared to HIP'ed samples

EFFECT OF SUBSTRATE POWDER PURITY, PRE-ALLOYED POWDER

Charpy impact tests:



Tensile tests:



HCR samples showed a higher USE and lower DBTT (-24° C) values than their elemental counterparts (-8° C), whereas although the USE also improved in the case of the prealloyed as-HIPed samples, the DBTT was in that case worse (+59° C) than for the elemental ones (+8° C).

	ROT: 0%			ROT: 65%		
Test T (° C)	25	450	750	25	450	750
R_m (MPa)	1085	792	260	718	384	203
$R_{p0.2}$ (MPa)	848	712	233	412	329	168
ε	0.097	0.081	0.050	0.16	0.095	0.04
ε_u	0.018	0.029	0.022	0.011	0.021	0.013

CONCLUSIONS

- Precipitation strengthening by fine oxide particles and transformation induced stress are the main cause of high tensile strength and stiffness of the as-HIPped ODS ferritic steels

- Larger oxides and nitrides at the pre-particle boundaries lead to lower fracture toughness and to brittle fracture

- Multiple hot cross rolling enhances the plasticity by decrease of the remnant porosity but also by an extensive structure recovery

- The Charpy tests showed a significant reduction of DBTT and an increase of the upper shelf energy when the deformation was 65% of thickness or higher

- The tensile test in all hot rolled steel samples showed a decrease in tensile strength and yield stress along with increase of ultimate plastic strain

- An additional improvement of plasticity was achieved by using the pre-alloyed powder instead of a mixture of elemental powders.

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USA, San-Diego,
9 - 13 October, 2012

FTP/4-5Rb:

LOW ACTIVATION VANADIUM ALLOYS FOR FUSION POWER REACTORS –THE RF RESULTS

V.M.Chernov, M.M.Potapenko, V.A.Drobyshev, D.A.Blokhin, N.I.Budylkin, E.G.Mironova, N.A.Degtyarev, I.N.Izmalkov, A.N.Tyumentsev, I.A.Ditenberg, K.V.Grinyaev, B.K. Kardashev, A.I.Blokhin, N.A.Demin, N.I.Loginov, V.A.Romanov, A.B.Sivak, P.A.Sivak, S.G.Psakhie, K.P.Zolnikov

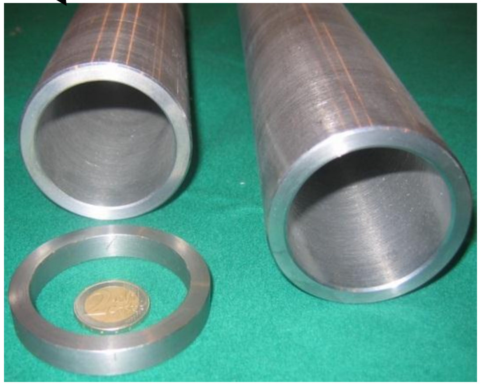
Bochvar High Technology Research Institute of Inorganic Materials (JSC “VNIINM”), Moscow, Russia,
Tomsk State University, Tomsk, Russia,
Ioffe Physical-Technical Institute, RAS, S.-Petersburg, 194021, Russia,
Leypunsky Institute of Physics and Power Engineering, Obninsk, Russia,
NRC “Kurchatov Institute”, Moscow, Russia ,
Institute of Physics Strength and Material Sciense, SB RAS, Tomsk, Russia

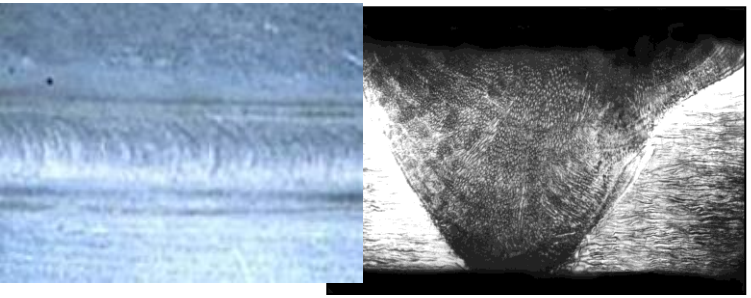
The RF vanadium alloys: Heats and articles (JSC “VNIINM”)
Referenced alloy V-4Ti-4Cr, Advanced alloys V-Cr-W-Zr

2010-2011, V-Cr-W-Zr
V-(4-9)Cr-(0.1-8)W-(1-2)Zr
heats of 0.5-2 kg,

2009-2011. V-4Ti-4Cr
heats: 100-110 kg

<2014.V-4Ti-4Cr
heats: 300 kg





welds (plates 2-6 mm)

- **plates up to 1930x367x15 mm, 1500x257x80 mm,**
- **tubes up to 67x6 mm**

VANADIUM ALLOYS - CHEMICAL COMPOSITIONS.

Alloy	CHEMICAL COMPOSITION (weight %)						
	Ti	Cr	W	Zr	C	O	N
V-4Ti-4Cr (VV1)	4.21	4.36			0.013	0.02	0.01
V-Cr-Zr		8.75		1.17	0.01	0.02	0.01
V-Cr-W-Zr		4.23	7.56	1.69	0.02	0.02	0.01

VANADIUM ALLOYS: V-4Ti-4Cr, V-Cr-Zr-C, V-Cr-W-Zr-C: Thermo-Mechanical Treatment (TMT) and Chemico-Thermal Treatment (CTT).

Promising ways to improve high-temperature strength-corrosion-radiation resistance are the methods of the TMTs and the CTTs using the combined methods of formation and modification of heterophase and defect substructures:

1. **The uniform distribution of the stable phases nanoparticles during $V_XC \rightarrow$ TiV (C, O, N) and $V_XC \rightarrow$ ZrC transformations by changing (controlling) mechanism of such transformations – from "*in situ* transformation" to the mechanism of dissolution of V_XC phase, followed by separation of fine carbides TiV (C, O, N) or ZrC from a supersaturated solid solution.**
2. **Microcrystalline structure under using of large plastic deformation in the intermediate stages of TMT and formation of defect substructures with high stored energy of deformation.**
3. **Ultra-fine particles of ZrO2 (CTT) in low-temperature diffusion alloying of oxygen (internal oxidation) which have a higher thermal stability and provide a significant (200 – 300 deg.) increase of the recrystallization temperature of alloys.**
4. **Structural states with both dispersed and substructure (by the elements of the dislocation, polygonal or microcrystalline structure) hardenings (TMT, CTT, TMT+CTT).**

VANADIUM ALLOYS: THERMO-MECHANICAL (TMT) AND CHEMICO-THERMAL (CTT) TREATMENTS

0. TMT-0: as received plates, roads and tubes (JSC “VNIINM”).

1. TMT-I : TMT-0 + annealing at (1000 – 1100) ºC, (40 – 60) min (vac).

2. TMT-II : TMT-I + annealing 1400 ºC (vac), 1h, + 3 cycles “deformation 30 – 50 % at RT, annealing at (600 – 700) ºC, 1h (vac)” + deformation (30-50) % at RT and annealing at (950 – 1100) ºC, 1h (vac).

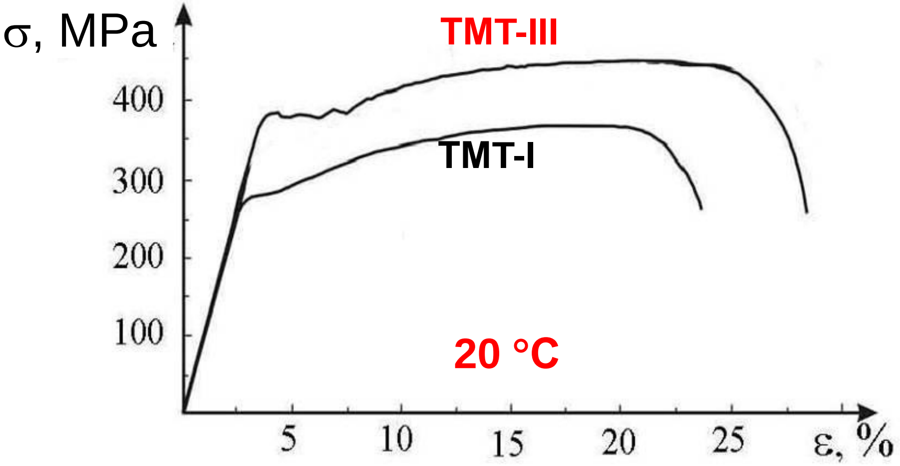
3. TMT-III : TMT-I + annealing 1400 ºC, 1h (vac) + 3 cycles “deformation 30 % at RT and annealing at 600 ºC, 1h (vac)” + 16 cycles with the changing of the deformation axis after each cycle “deformation 30 % at RT and annealing at 1000 ºC, 1 h (vac)”.

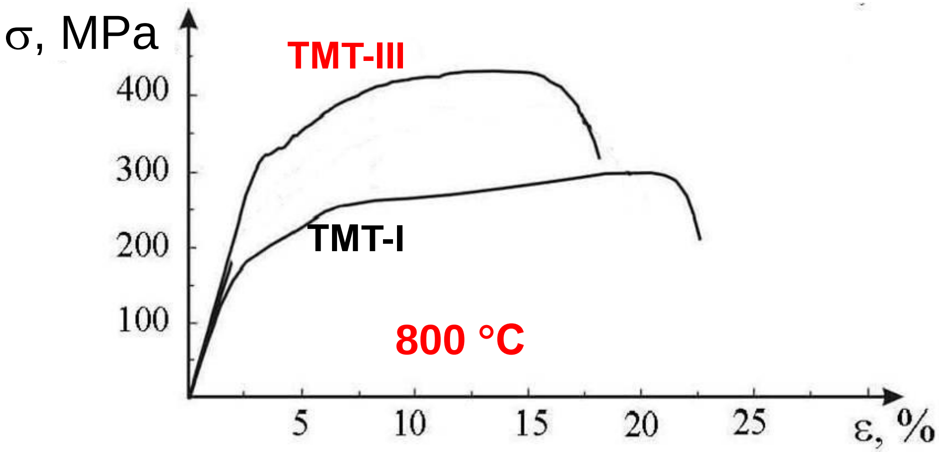
4. TMT-IV: TMT-I + annealing 1400 ºC (vac), 1h, + 3 cycles with the changing of the deformation axis after each cycle “deformation 30 % at RT and annealing at 600 ºC, 1 h (vac)” + 16 cycles with the changing of the deformation axis after each cycle “deformation 30 % at RT and annealing at 900 ºC, 1 h (vac)”.

5. CTT-I (Chemico-Thermal Treatment with oxygen saturation of the alloy): TMT-0 + annealing at ≈ 600 ºC, ≈1h (air, oxidation saturation) + annealing at (800 – 1200) ºC, (1 – 2) h (vac). Annealing time and temperature are depended from the final oxygen concentration in alloy.

6. CTT-II: TMT-II + CTT-I.

V-4Ti-4Cr: TMT-I AND TMT-III. MECHANICAL PROPERTIES





TMT mode	$\sigma_{0.1}$, MPa	δ, %	ψ, %
Testing temperature 20 °C			
TMT-I	290-300	19-20	80-91
TMT-III	370-380	23-24	83-87
Testing temperature 800 °C			
TMT- I	170-180	17-19	81-86
TMT-III	270-280	13-15	75-85

Structure and phase modifications (TMT-III) lead to a significant increase in the strength of the alloy in a wide temperature range (up to 800 ºC).

The absolute value of hardening ($\Delta\sigma \approx$ 100 MPa) is weakly dependent on temperature.

V-4Ti-4Cr: TMT-I – TMT-IV. MECHANICAL PROPERTIES

TMT	Method of modifying the microstructure	$\sigma_{0.1}$, MPa	δ, %	ψ, %
Test T = 20 °C				
TMT I	The standard treatment regime	290-310	19-20	80-91
TMT II	Change of the mechanism of $V_2C \rightarrow TiV(C,O,N)$ transformation	330-340	20-25	85-90
TMT III	The formation of a more small microcrystalline structure	370-380	23-24	83-87
TMT IV	Extremely high dispersity of second phase particles and substructures with a high density of defects.	390-420	15-17	
Test T = 800 °C				
TMT I	The standard treatment regime	170-190	17-19	81-86
TMT II	Change of the mechanism of $V_2C \rightarrow TiV(C,O,N)$ transformation	210-230	17-18	76-80
TMT III	The formation of a more small microcrystalline structure	270-280	13-15	75-85
TMT IV	Extremely high dispersion of second phase particles and substructures with a high density of defects.	330-370	13-14	

VANADIUM ALLOYS: V-Cr-Zr-C and V-Cr-W-Zr-C: TMT-I, CTT-II. Mechanical properties

TMT	**Test T = 20 °C**			**Test T = 800 °C**		
	$\sigma_{0,1}$, MPa	σ_B, MPa	δ, %	$\sigma_{0,1}$, MPa	σ_B, MPa	δ, %
V-Cr-Zr-C						
TMT-I	**240**	**395**	**25**	**180**	**235**	**26**
CTT-II	**730**	**840**	**6.5**	**370**	**400**	**8**
V-Cr-W-Zr-C						
TMT-I	**300**	**480**	**25**	**190**	**265**	**25**
CTT-II	**675**	**810**	**4.5**	**400**	**425**	**6.5**

CONCLUSION: The RF Low Activation Vanadium Alloys for Nuclear Fusion and Fission Reactors Applications (coolants Li, Na, Pb, Pb-Li).

Potential of recycling vanadium alloys can make structure waste manageable.

VANADIUM ALLOYS ARE THE REAL ALTERNATIVE TO ALL TYPES OF FERRITIC-MARTENSITIC STEELS.

<2012: Referenced alloy V-4Ti-4Cr: Heats up to 110 kg. Any articles.
Recommendations for the nuclear applications:
<100 dpa-Fe, T-window (300)350 – 750(800) °C.
Applications: TBM DEMO in ITER, DEMO-FusionPowerPlant (Li, Pb-Li),
FastBreederReactors: BN-1200(Na), MBIR(Na), BREST (Pb).

The alloy V-4Ti-4Cr is the best alloy of the V-Ti-Cr system (USA, Japan, Russia).
The RF Knowledge Data Bases seem to be appropriate for the V-4Ti-4Cr alloy but further progress is anticipated for the advanced alloys of the system V-Cr-W-Zr-C-O.

< 2020: OPTIMISM UP TO 160 dpa-Fe, T-window <300 – 850(900) °C.

REFERENCED V-4Ti-4Cr: Large heats (150-300 kg) and articles. Optimization (minimization) of the technological concentration of impurities.

Reactor tests: BN-600, 10-160 dpa-Fe, Tirr = 380 °C – 700 °C.

Corrosion tests (Li, Na, Pb, Pb-Li).

ADVANCED V-Cr-W-Zr-C-O (heats up to 40 kg):

- further optimizations of chemical compositions and regimes of thermal-mechanical-chemical treatments (TMT&CTT) of heats and articles, higher the thermal stability of solid solutions, nanoparticles, substructures and grain boundaries,
- heats, articles and reactor properties.