Neutron as a daily tool towards the application to the steel industry

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in collaboration with
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"CANS is usuful for pre-experiment & education." *Only for them?*

Compact source is spreading in Japan

JCANS

Hokkaido Univ.
from 1973

Tohoku Universit, planning

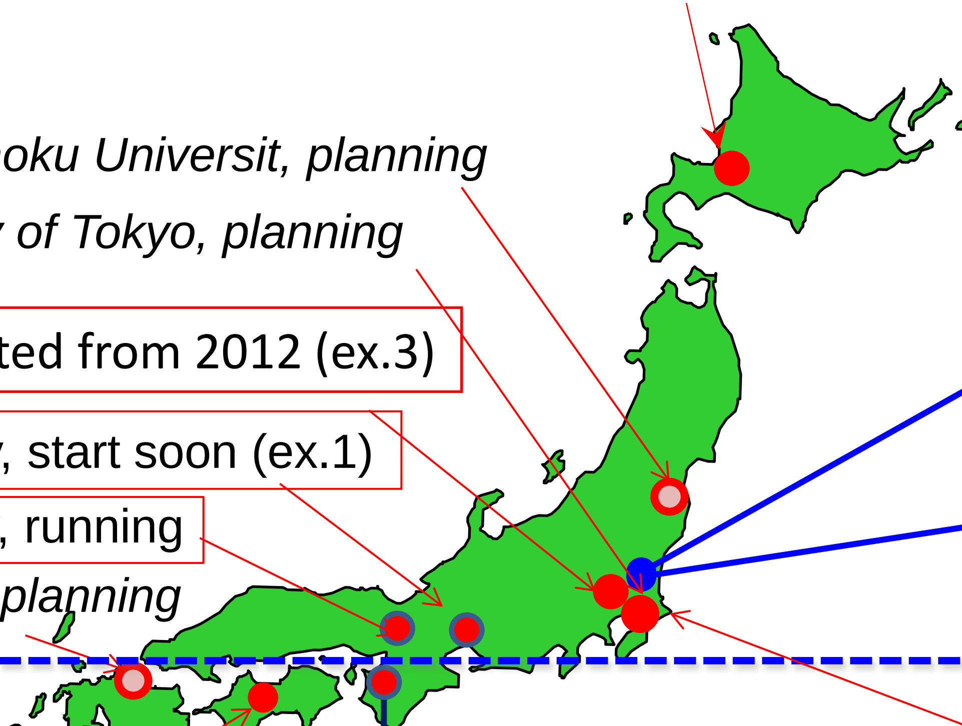
University of Tokyo, planning

RIKEN : started from 2012 (ex.3)

Nagoya University, start soon (ex.1)

Kyoto university, running

Kyushu university, planning



large facilities

J-PARC

reactor:JRR-3

Ibaraki CANS start soon (ex.2)

Sumijyu kensa
(private company)

Kyoto university research reactor **KURRI**

CANS activity in Japan ~ 1st example, Nagoya Univ.

NUANS：Engineering test of BNCT & Engineering and science applications

1）BNCT engineering test

Electrostatic Acc.
（IBA 2.8MeV, 42kW)

Sealed Li target

↓

Compact and low radioactive system

↓

Engineering feasibility of the BNCT system

2）Science・Engineering applications：Be target

Imaging (Power＜4kW)

project leader Profs Shimizu & Kiyanagi

NUANS
Nagoya University Accelerator-driven Neutron Source

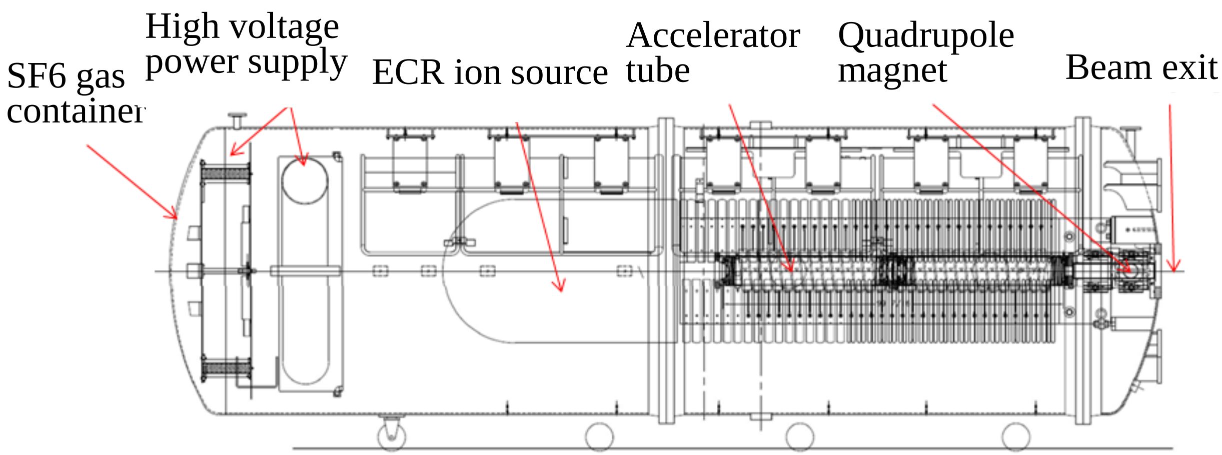
CANS activity in Japan ~ 1st example, Nagoya Univ.

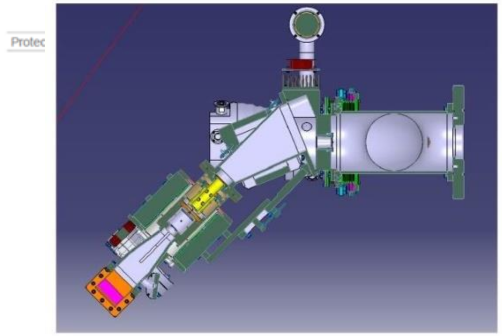
IBA Dynamitron accelerator

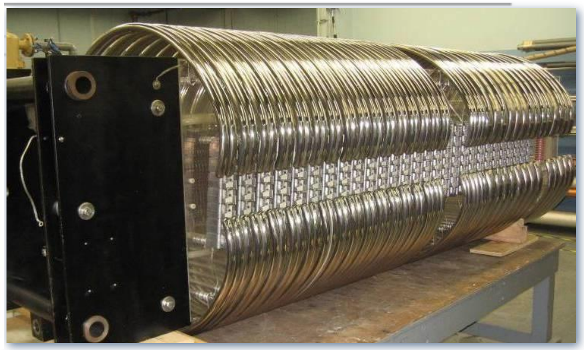
Ep : 1.9–2.8 MeV (variable)

Ip : 15mA 、DC

Size and weight : 7.5 m × 2.8 m、6.5 ton







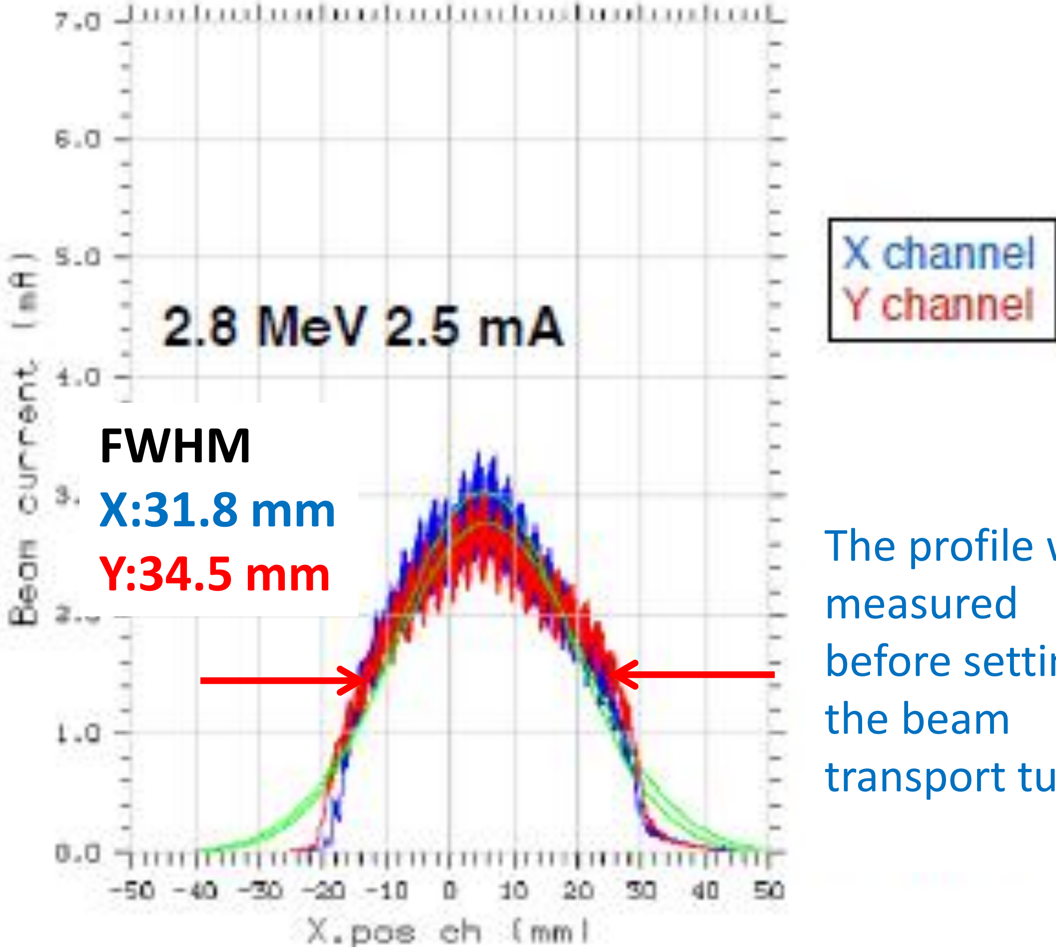
NUANS
Nagoya University Accelerator-driven Neutron Source

Beam profile observed during commissioning

January of 2016

We got 2.8MeV, 11mA beam (~70%)

Commissioning is continuing for stable operation



The profile was measured before setting the beam transport tube.

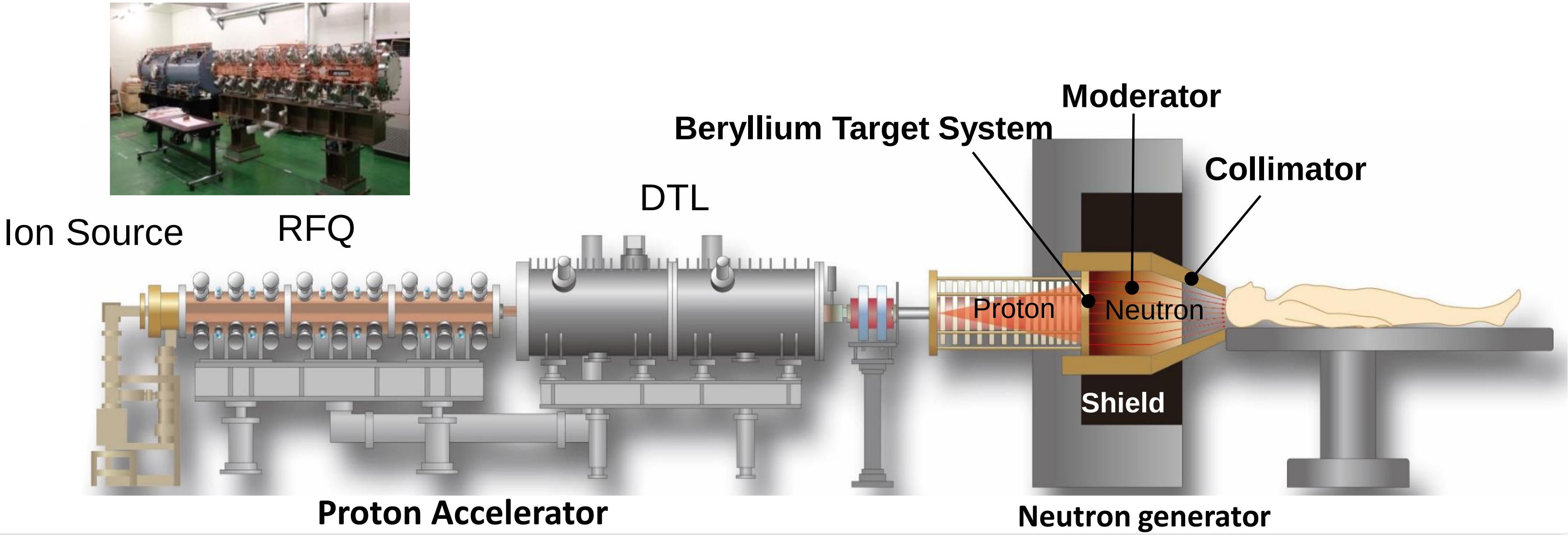
CANS activity in Japan ~ 2nd example, Ibaraki Pref.

Project Team : Univ. of Tsukuba, KEK, JAEA, Hokkaido Univ., Ibaraki Pref., Mitsubishi Heavy Industry, etc.

Concept : Realization of BNCT with Safety, Stable & Easy in a Hospital

Research & Development;

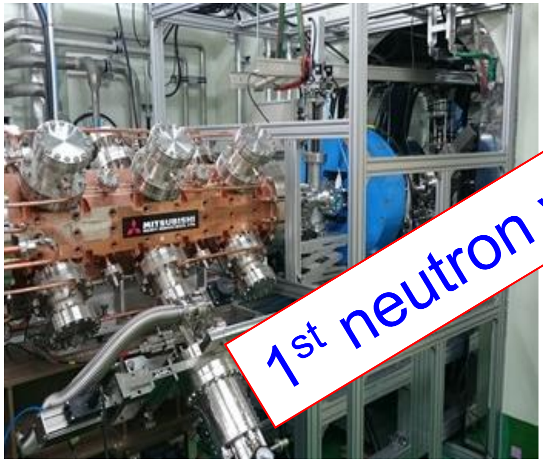
- **Compact & high power proton accelerator**
- **Neutron generator with neutron target device, moderator, Collimator and Shield applicable to NCT treatment.**
- **Treatment planning system, patient setting, neutron monitor & PG-SPECT.**



CANS activity in Japan ~ 2nd example, Ibaraki Pref.

RFQ + DTL Type Linac

+



Ion Source + LEBT

1^{st} neutron will come soon

Klystron

Type	RFQ＋DTL Type Linac
Proton Energy	8MeV
Peak Current	50mA
Average Current	>5mA （Max.10mA）
Beam plus	1msec.
Duty	20%
Power to Target	>40kW （Max. 80kW）
Dimension	Length: <7m, Footprint: <50m^2

CANS activity in Japan ~ 3rd example, RIKEN, already in use

RANS (RIKEN Accelerator-driven compact neutron source)

compact neutron source for practical use

Proton linac, (commercially sold accelerator) (1.5 M.US$)

Ep=7MeV

Ip<100 μA maximum averaged current

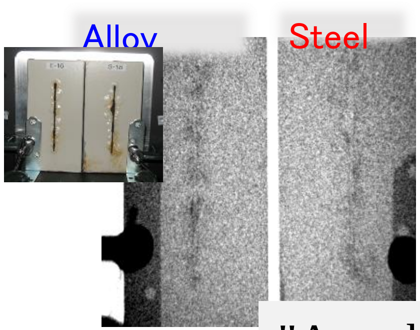
Δτ : 10-180μs pulse width of proton (30μs→modified)

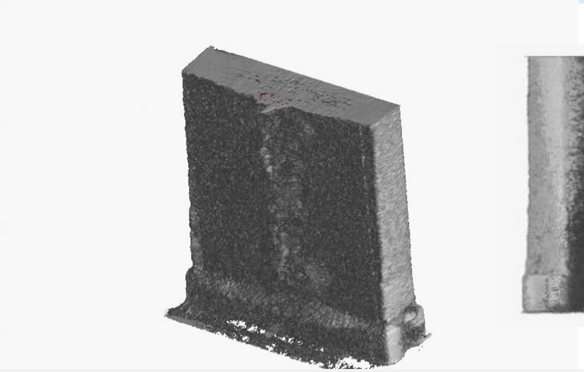
Fr : 20-180Hz repetition rate of proton

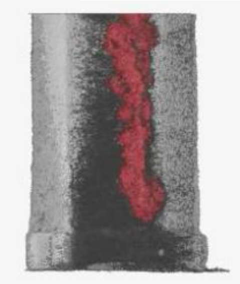
project leader is Dr. Otake

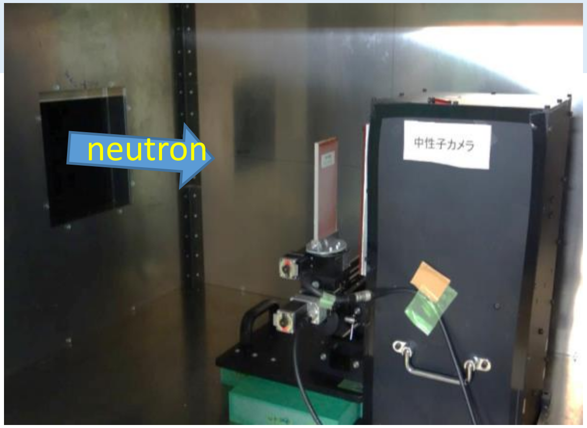
1. Industrial use –iron and steel-

A) Imaging : Corrosion and water movement









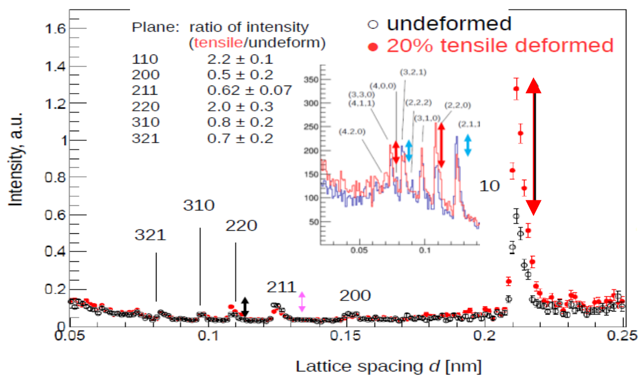
"Atsushi Taketani, et al : ISIJ International Vol. 57, No. 1 (2017)

RANS (RIKEN Accelerator-driven compact neutron source)

compact neutron source for practical use

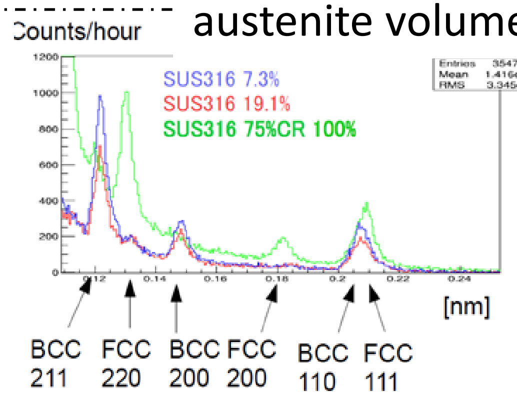
B)Engineering diffraction：texture evolution, austenite volume fraction

texture evolution



2minutes each diffraction measurements

austenite volume fraction

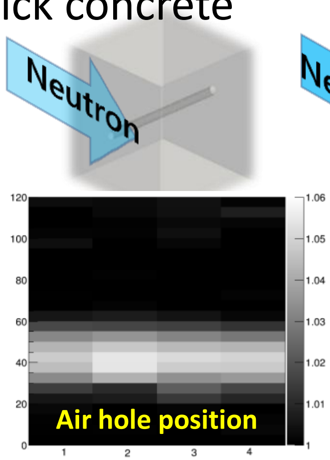


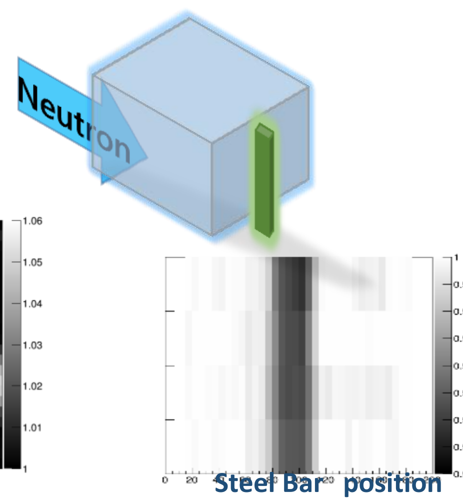
volume fraction from RANS diffraction	Volume fraction from the size
6.7±0.8%	8.3%
17.4±0.8%	19.1%

Y. Ikeda, et al Nucl. Instr. Meth. A833 (2016) 61-67

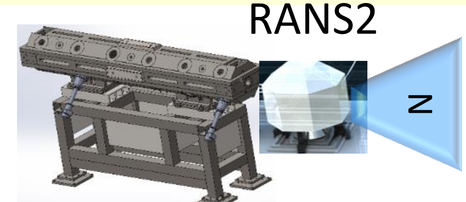
2. Social safety- Non-destructive inspection for social infrastructures, bridges, roads

Success of the observation of air hole and steel bar position through thick concrete





Accelerator for RANS2 is coming Jan 2017
2.49MeV proton



Neutron as a daily tool
towards the application to the steel industry

CANS itself is useful and good enough for promoting materials science

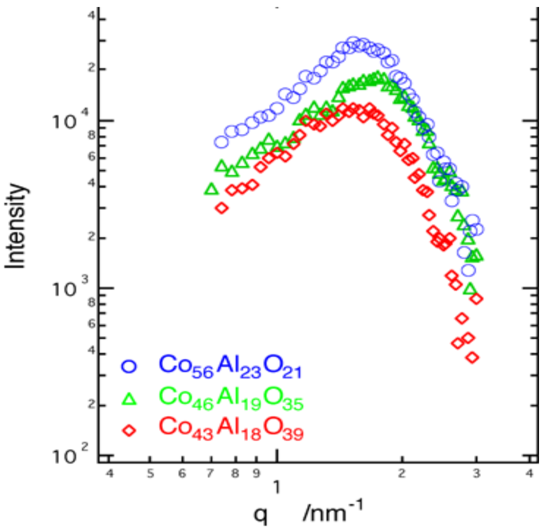
1. "Unique information"
   accurate evaluation of main constituent element in nano-precipitates
   by Combined method of SAXS and SANS

2. "Easy & Quick": daily use of neutron as the first step of characterization
   by in-house compact neutron source

we are in " nano-tech age" ! high-Q region in SAS is important

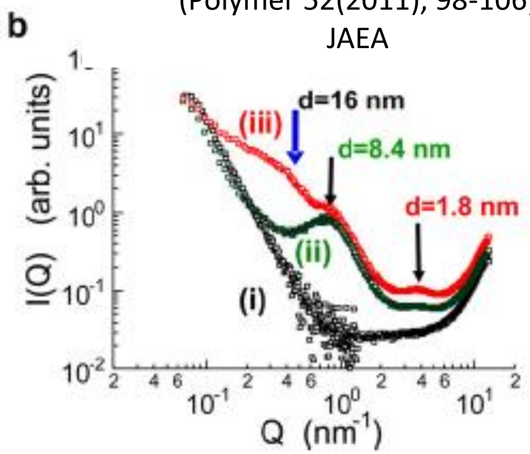
magnetic field sensor

(J.Appl.Phys. 82(1997),5642-5646
Tohoku Univ.



Fuel cell

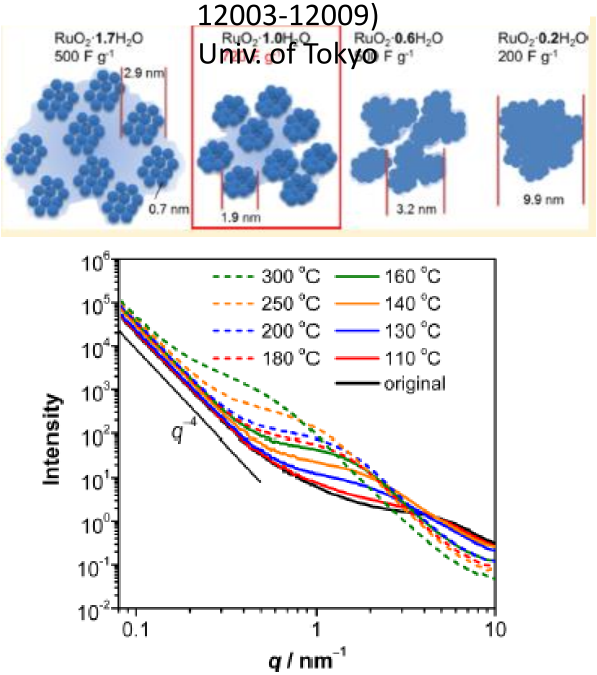
(Polymer 52(2011), 98-106)
JAEA



grafted nafion

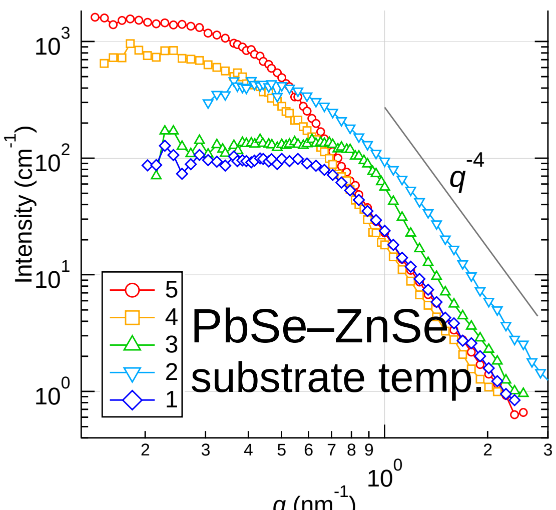
Capacirter (battery)

(J.Phys.Chem.C, 117(2013), 12003-12009)
Univ. of Tokyo



Solar cell

(J.Phys D:Appl.Phys. 47(2014), #435102)
RIEM



but today, focus on the steels as examples

Why steel, too old? Structural Materials are still under developing !

constructed in 1887 – 1889
height 324m
7300 t (wrought iron!)
160~220N/mm^2
0.05~0.25wt%C

1957 – 1958
height 333m
4000 t (steel)
240N/mm^2
0.3~2wt%C

2008 – 2012
height 634m
32000 t
400N/mm^2
700N/mm^2 (gain tower)
Mn, Ni, Mo addition ~0.1%C

microstructure control by alloying and processing

Quantitative Evaluation of microstructure is important !
example of SAS application: Size and number density of oxide in ODS steels

Acta Materia., 57, 5571-5581(2009)

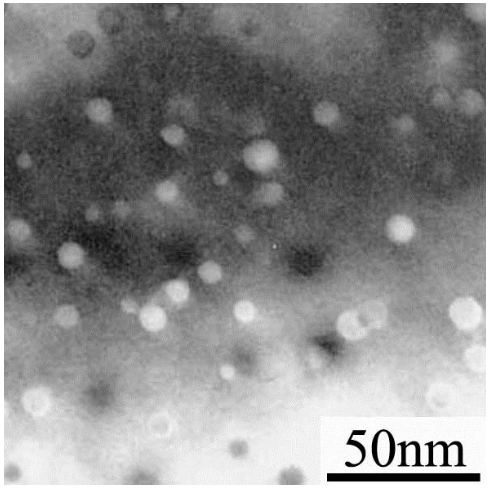
9wt%Cr-0.13C-0.35Y_2O_3-(0.2~0.4)Ti-(1~2.4)W-(0.08~0.15)ex.O

cladding tube for fast breeding reactor

feature

low swelling rate

high creep strength



konwn

relation between composition and propeties

structure of oxide is $Y_2Ti_2O_7$

unknown

size and number density

composition of $Y_2Ti_2O_7$ (composition of Fe)

Conventional use of SAS, size and number density of ODS steel

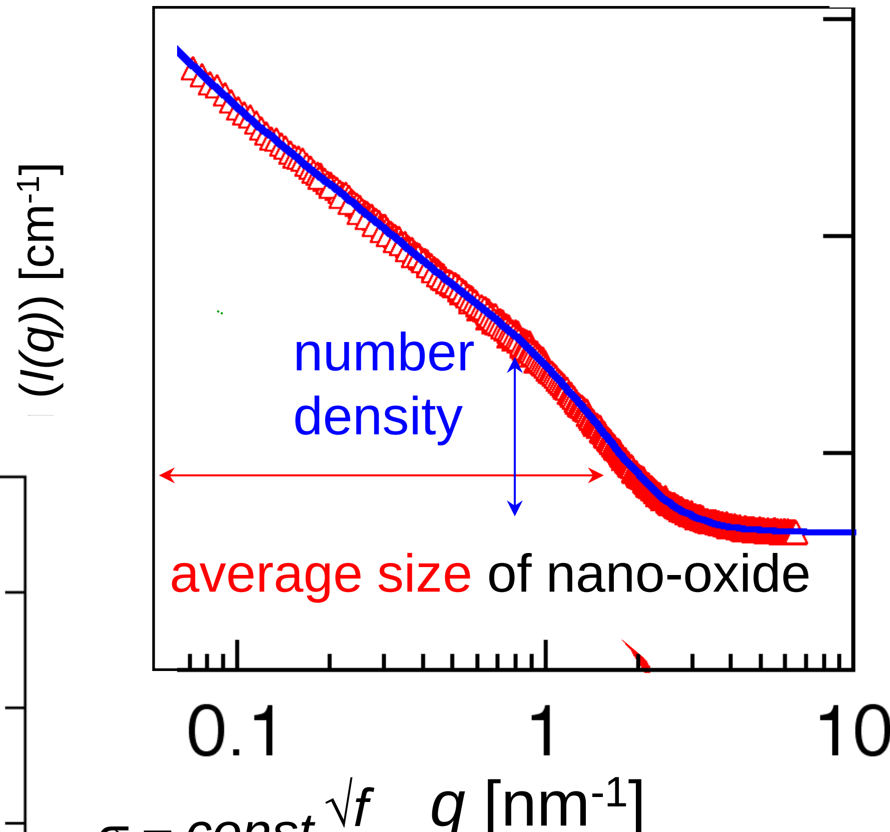
Intensity = $(\Delta\rho)^2 \cdot$ (number density) $\cdot$ volume^2 $\cdot$ (form factor)^2

intensity of profiles — width of the profile

$\Delta\rho$ depends on the phase *known !*

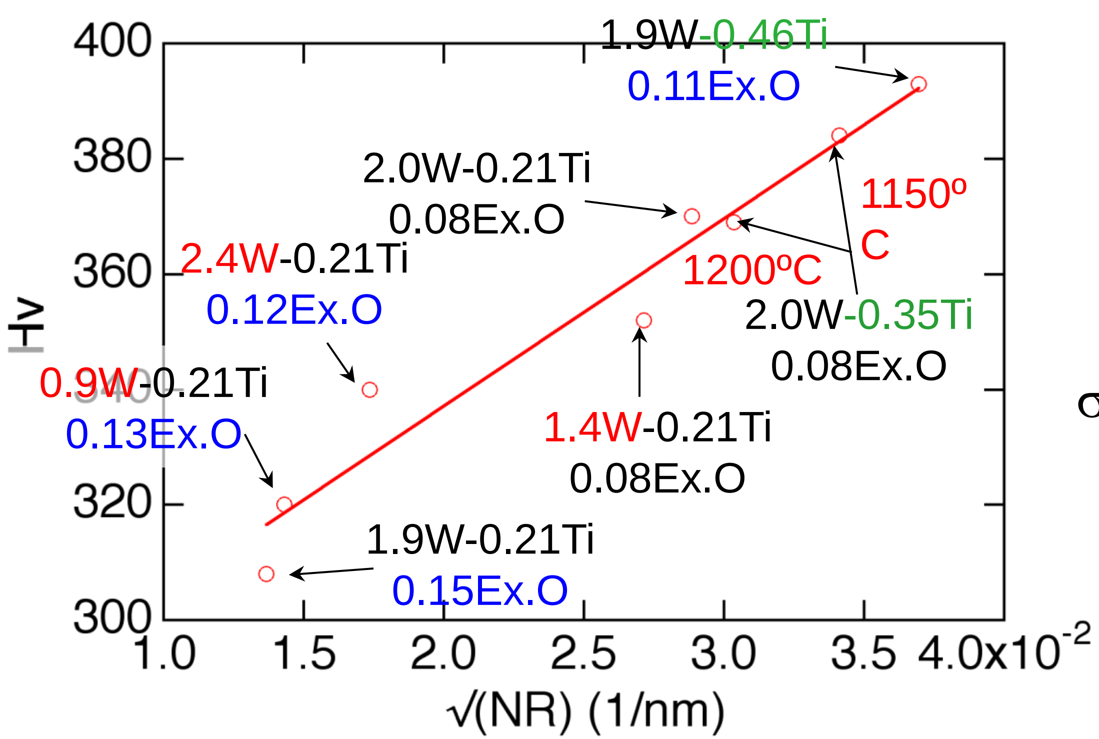
$$\Delta\rho_{Y2Ti2O7} = \rho_{Y2Ti2O7} - \rho_{matix}$$

size & number density



$$\sigma = const. \frac{\sqrt{f}}{R}$$

$$= const. \sqrt{(NR)}$$



new way to get compositional information: Combination of SAXS & SANS

intensity = $(\Delta\rho)^2$ x number density x (volume x form factor)^2

composition

determining factor of Intensity
one of them should be known !

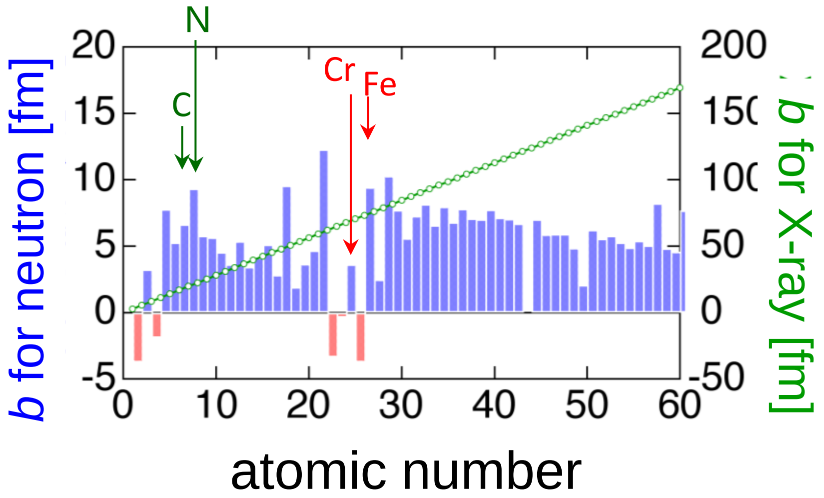
determined by microstructure
: no difference in SAXS and SANS

ratio between SAXS and SANS
→ cancelled out

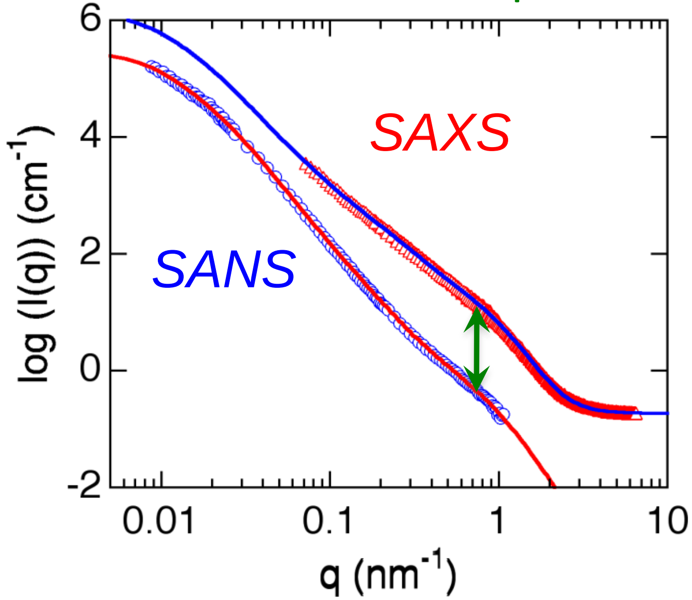
$$\Delta\rho = \rho - \rho_{\mathrm{matrix}} \qquad \rho = \sum_i n^a c_i^a b_i$$

n^a : number density of atom, c^a: atomic fraction,
b_i: scattering length

different dependence on atomic number !

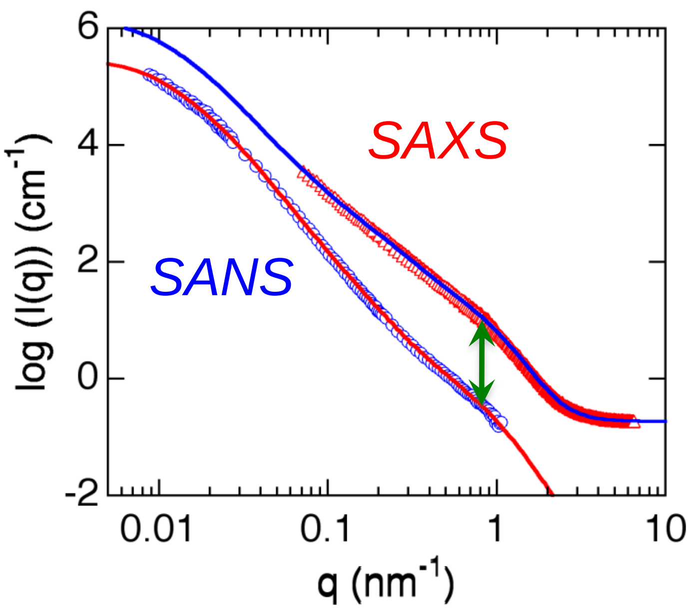


information of composition



Each of SAXS and SANS cannot determine composition, but together, they can !

Combined use of SAXS and SANS : accuracy is independent of size



way to use #1 : phase determination from candidate

8 different heat ODS steels

$I_{SAXS} / I_{SANS} = 40 \pm 4$

	$Cr_{23}C_6$	TiC	**$Y_2Ti_2O_7$**	Y_2TiO_5	Y_2O_3	Cr_2O_3
$\Delta\rho_{SAXS}^2/\Delta\rho_{SANS}^2$	4.6	16	**40**	48	60	69

way to use #2 : difference from equilibrium

substitute for Y

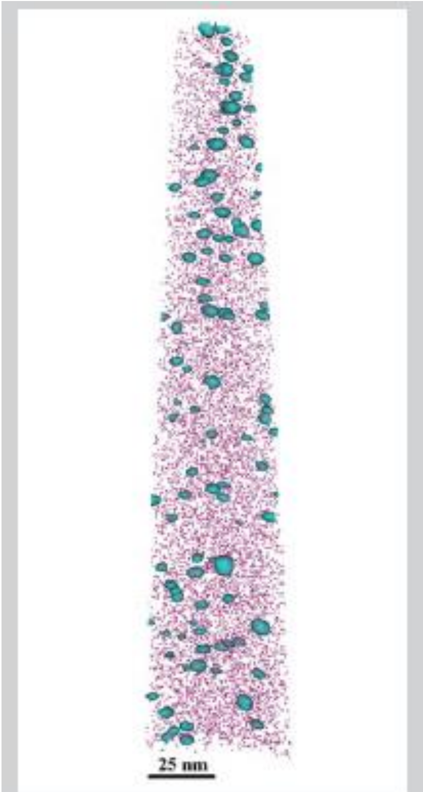
x < 0.2 for $(Y_{1-x}Fe_x)_2Ti_2O_7$; ~ 4at%

substitute for Ti

x < 0.15 for $Y_2(Ti_{1-x}Fe_x)_2O_7$; ~ 3at%

substitute for O

x < 0.1 for $Y_2Ti_2(O_{1-x}Fe_x)_7$; ~ 6at%



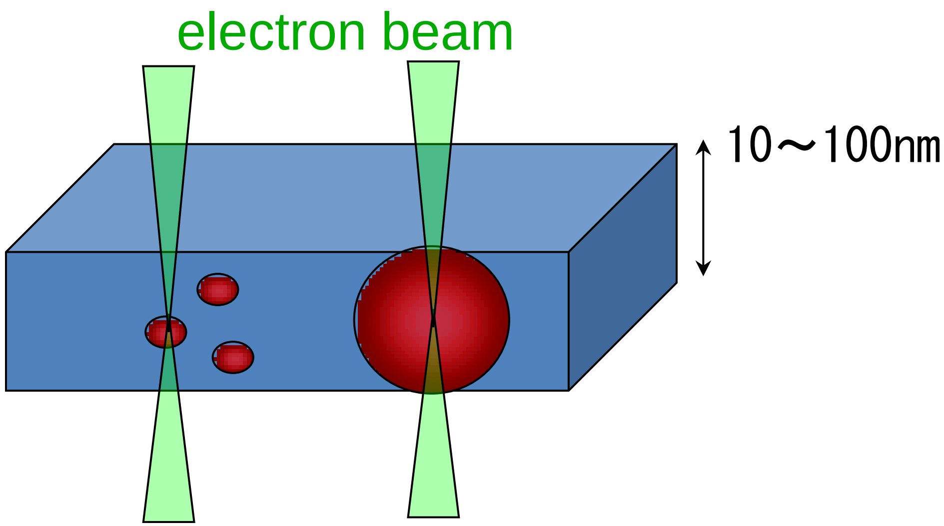
However, it is difficult to reconcile the SANS data with NFs that are so highly enriched in Ti that would result in much lower M/N than are observed.[12] APT results also indicate that the NFs contain large amounts of Fe (≈40 to 70%). However, the Fe is likely an APT artifact.[11,14] Varying amounts of

Odetta et al., JOM, 2010

How to characterize compositions of heterogeneities smaller than 1 nm

powerful for observing partition of alloying (minor) elements

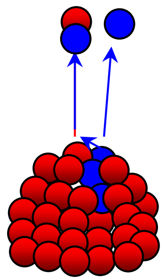
TEM-EDX



effect from matrix elements

accurate comp.

Atom Probe



atomic resolution (in depth)
evaporation aberration
↓
effect is large for main elements
below 1~2 nm heterogeneity

How to observe partition of main elements

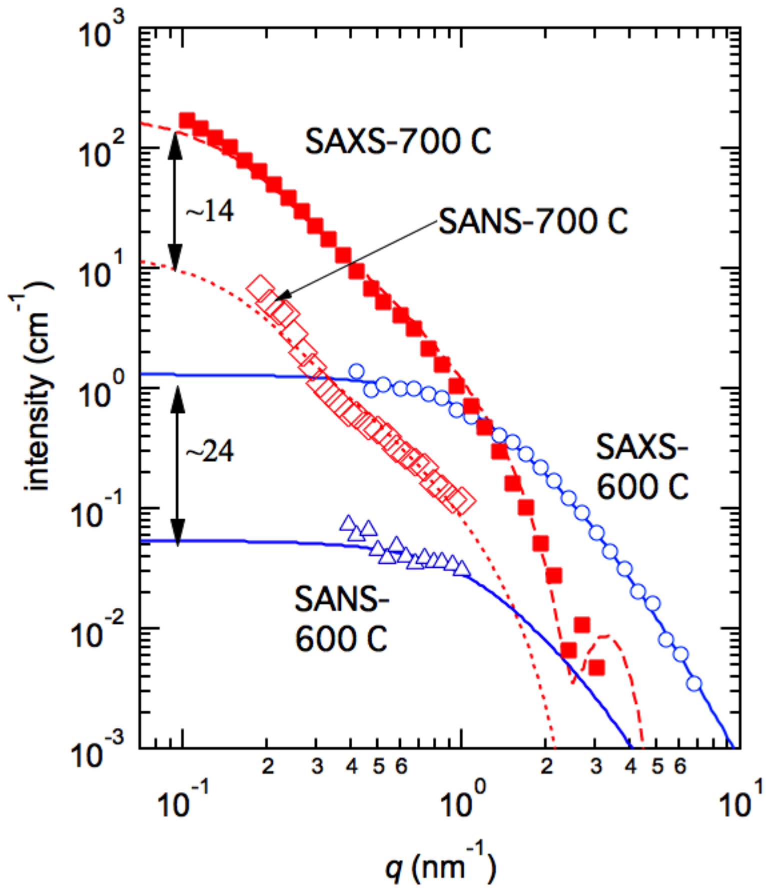
combined use of SANS & SAXS
sensitivity independent of size

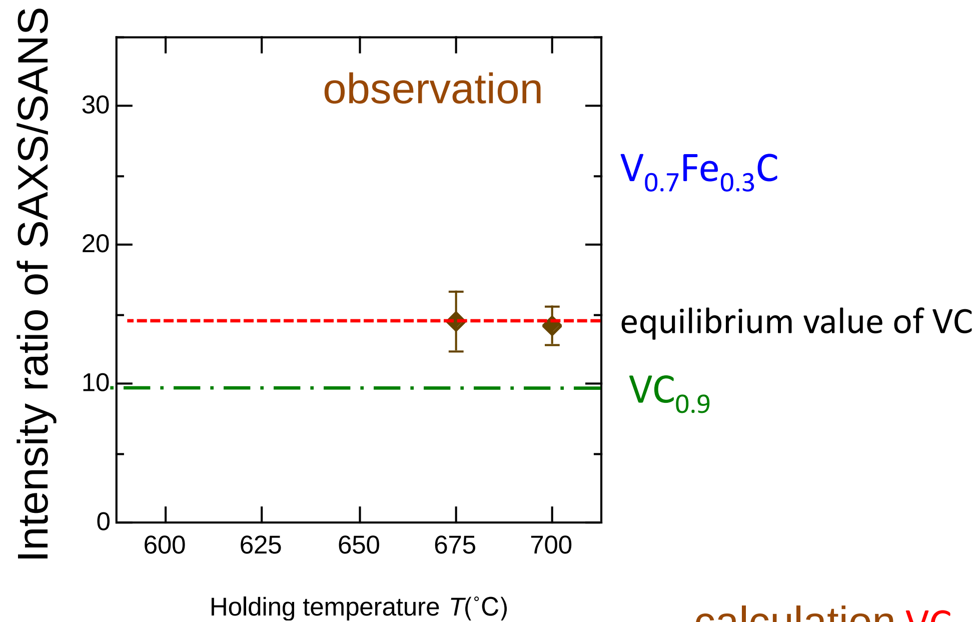
possible to discuss main elements（in this case, Fe)
No other techniques make it possible

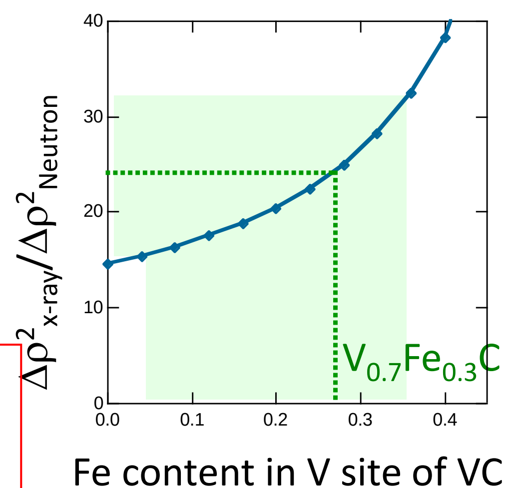
What happen in the early stage of precipitates

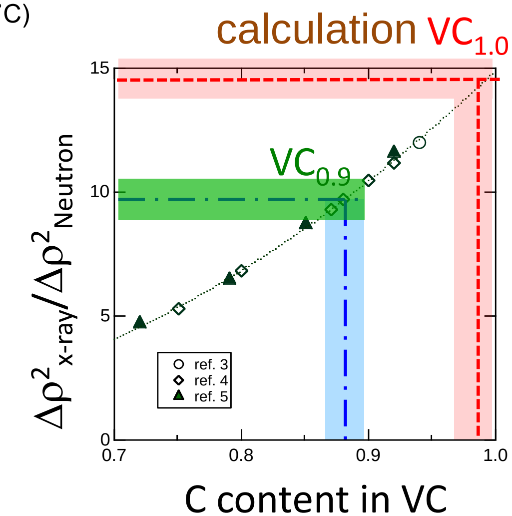
Y. Oba et al.,ISIJ International, 51, 1852-1858(2011)

Combined use of SANS and SAXS









1 nm diameter, a lot of Fe

↓

embryo (cluster)

→ contribute to the strength

towards easy and quick characterization of nano-structure

for SAXS measurements, anytime we can measure

NIMS labo-SAXS

q_{min} is only 0.1nm^{-1}

⟶ focus on nanostructure

20~100μm by Mo-Kα

SANS-J-II

for SANS measurements

twice a year or less than it.

writing good application is required

rate determining process

18m SANS

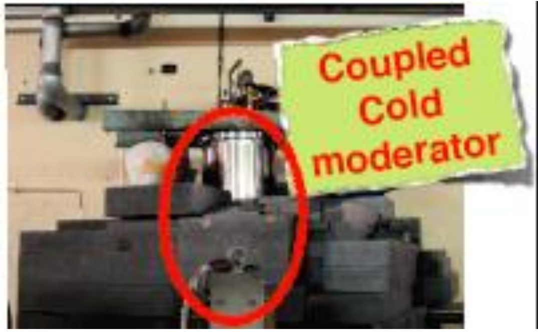
labo-scale SANS makes us faster...

45MeV Electron Linac based pulsed cold neutron source @Hokkaido University

Electron Linac

First beam: 1973

35 MeV, 30 μA, 50 pps : ~ 1 kW



Cold neutron source

W & Pb-Target

Solid methane cold moderator @17K

mainly use for development of target and moderator
too weak for scattering experiments.......

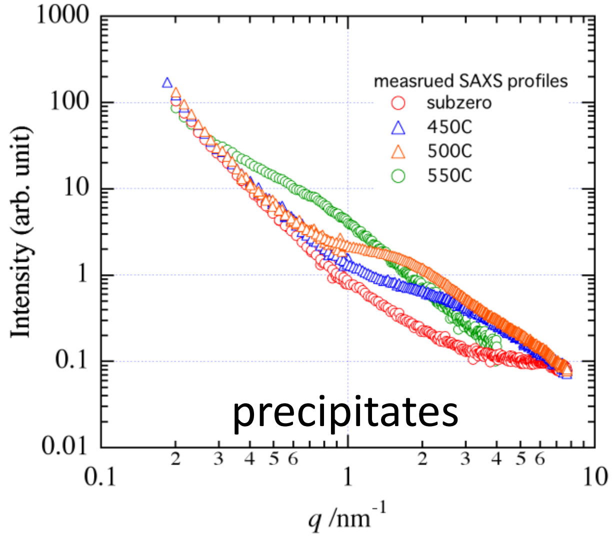
Can we get SANS signal in reasonable measurement time?

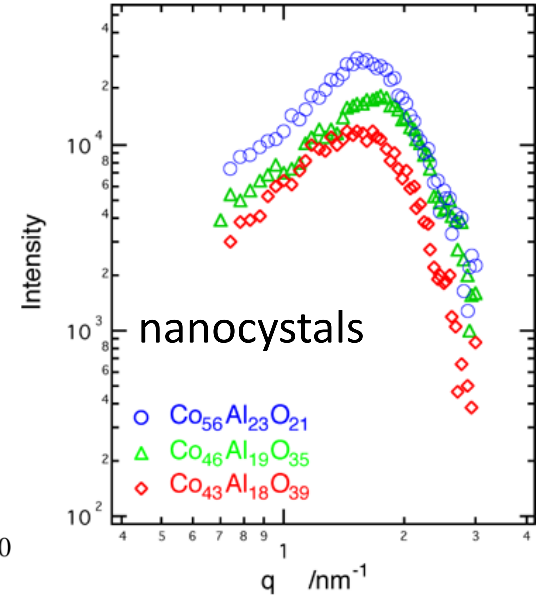
Why we can get comparable date to large facilities
→ optimize resolution to our purpose

Labo.Mo-SAXS (NIMS->Hokkaido Univ)

covered q-range

$0.2 < q < 10\ nm^{-1}$





intermediate-Angle Neutron Scattering

(iANS: "irons")

Target: $0.2 < Q < 10\ nm^{-1}$

conventional SANS		new SANS
2〜3mrad	→	10 mrad
0〜5°	→	5〜15°

Intensity of beam in use

$I(\Delta Q) \sim \Delta\theta^4$ x 600

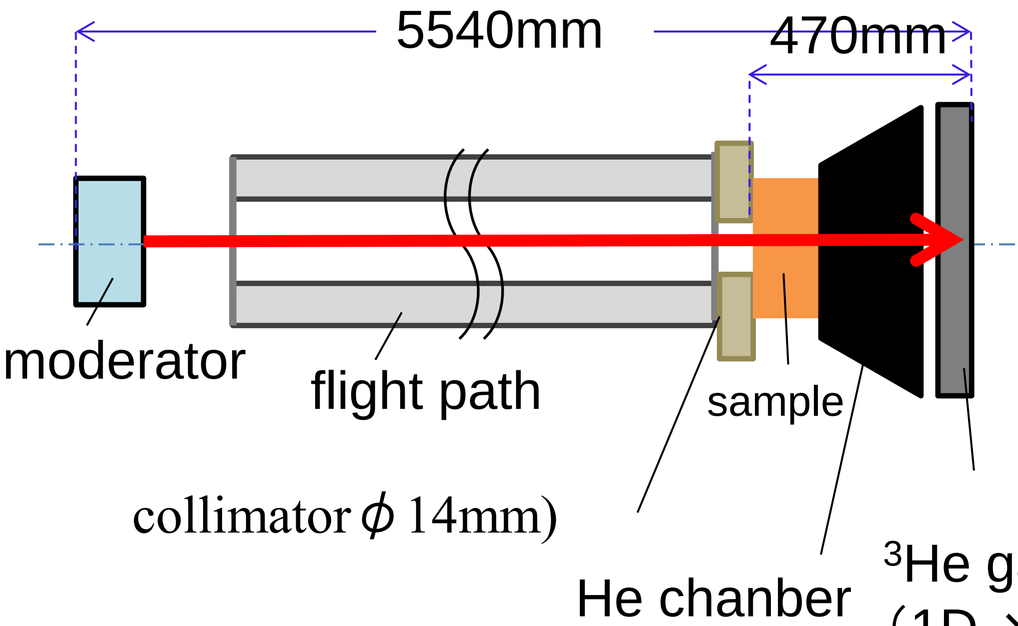
compact neutron

→ tailor to your purpose

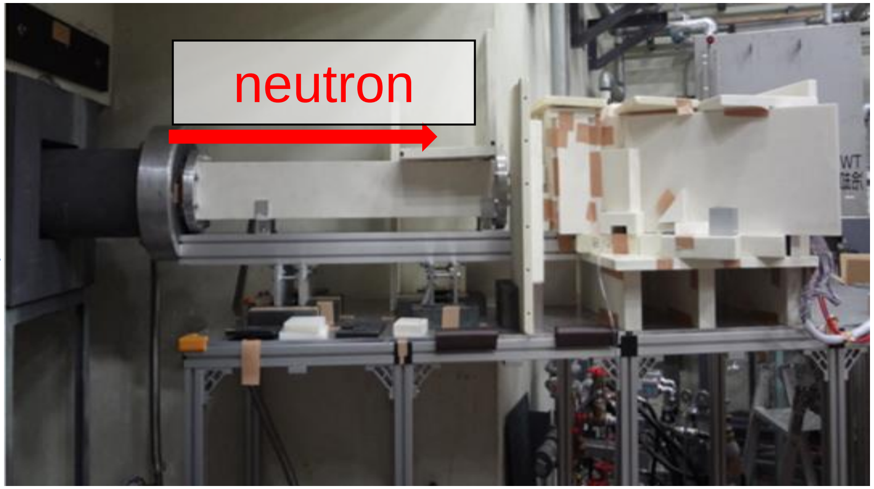
iANS（intermediate-Angle Neutron Scattering）

target q-rangt $0.2 < q < 10nm^{-1}$

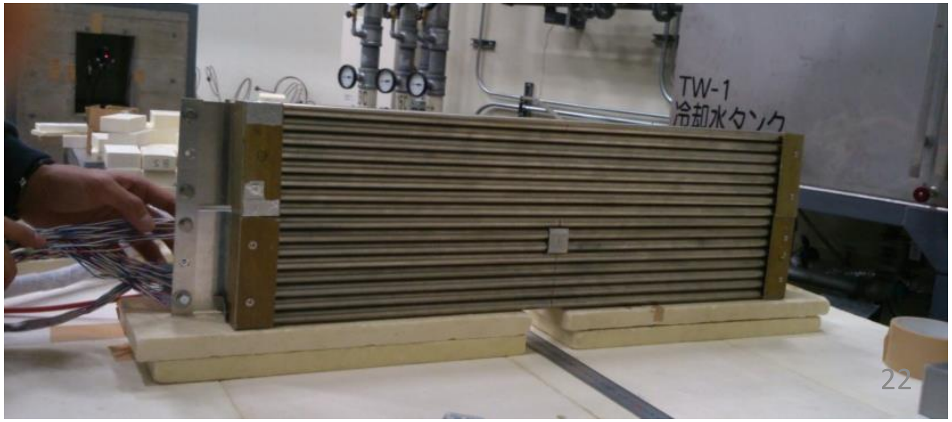
Schematic view of iANS（from top）



photos of iANS

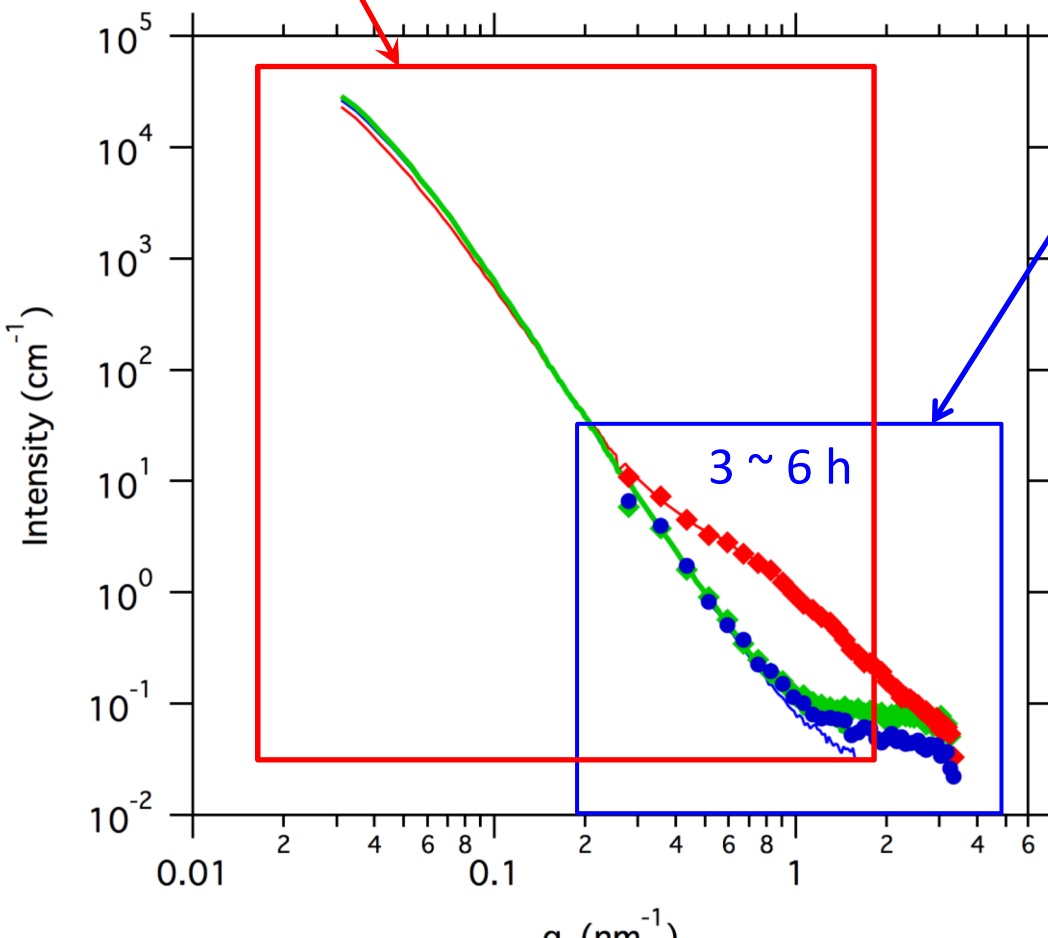


^{3}He gas detector
（1D × 16 tubes）



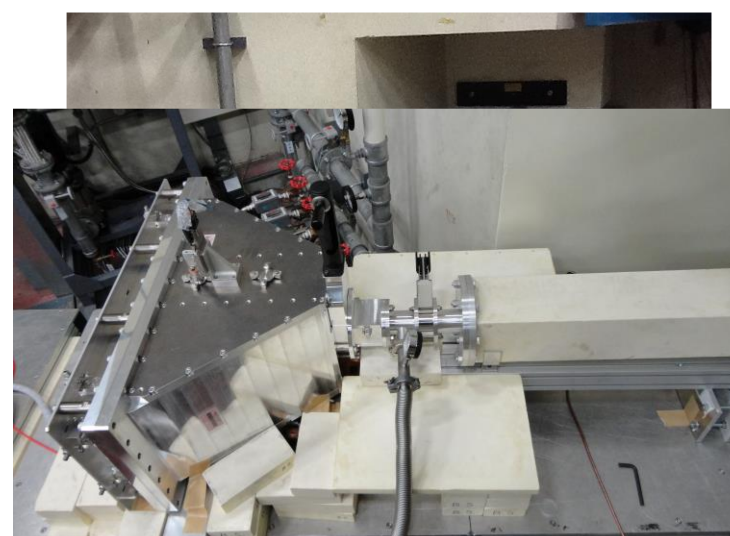
Compact Neutron source give data comparable to big facilities

18m SANS
cold neutron source in HANARO
(30 MW, λ=9.6, 4.8 A)
sample to detector 9m, 3m
$0.03 < q < 5 nm^{-1}$



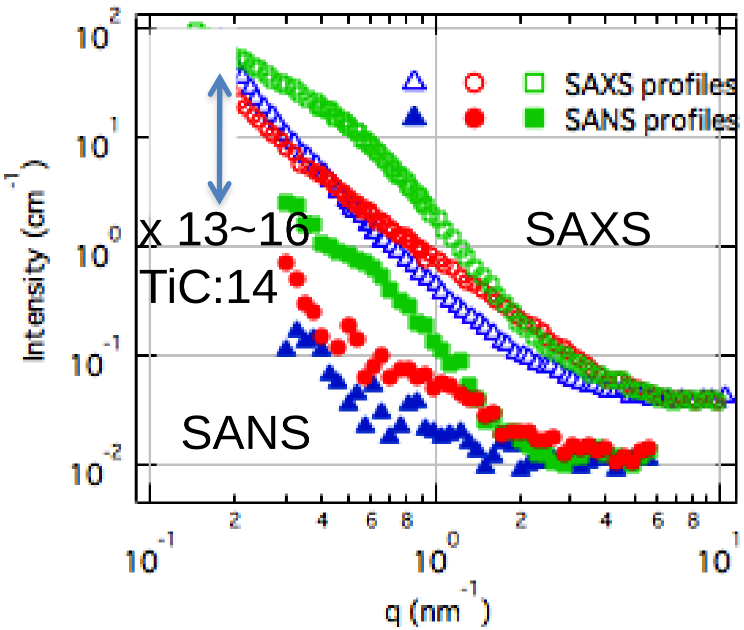
data from precipitate measured by iANS in HUNS

(1 kW, $4 < \lambda < 10$ A)
$0.1 < q < 10 nm^{-1}$

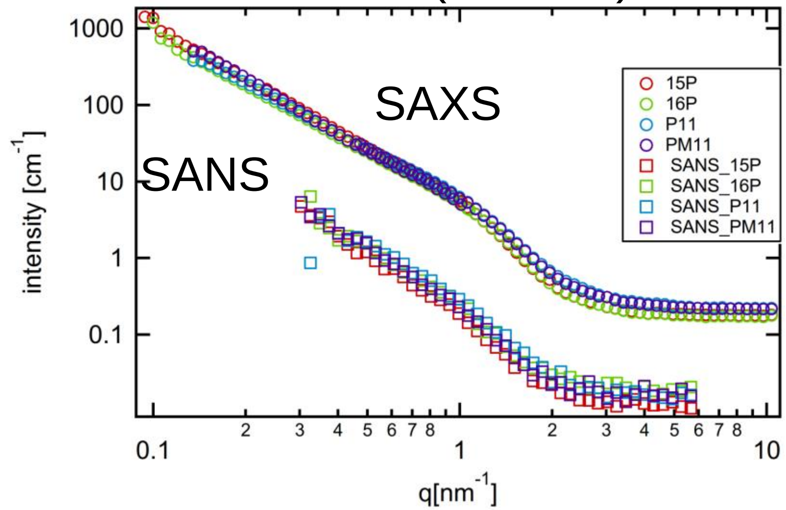


From Octorber in 2015, SANS monthly in Hokkaido.Univ.

TiC precipitates in steels (2015.10)

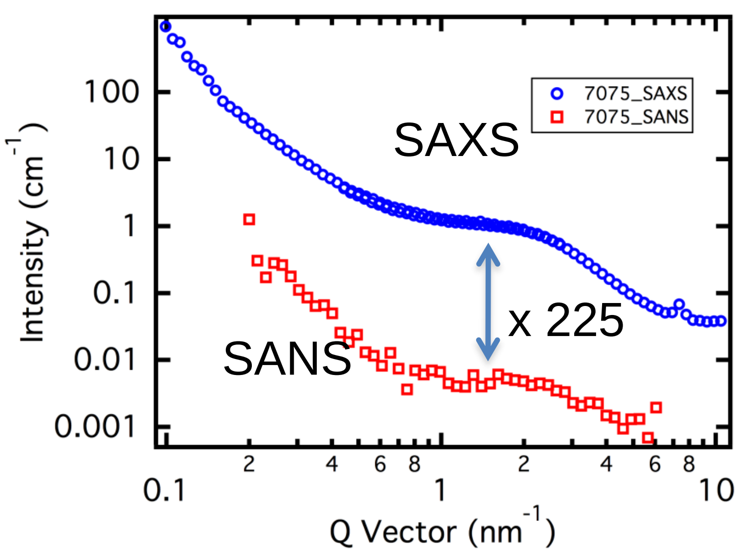


ODS steels (2015.11)



SAXS/SANS ratio can be evaluated only using in-house facilities

Al alloys：7000 series（2015.12)



$MgZn_2$:230

measurements time is still long
- SAXS: 3~10 h
- SANS: 6~8 h

Long measurement time is not so bad, you may enjoy it because...

Sapporo, München, Milwaukee

Neutron as a daily tool
towards the application to the steel industry

CANS itself is useful and good enough for promoting materials science

1. "Unique information"
   accurate evaluation of main constituent element in nano-precipitates
   by Combined method of SAXS and SANS

2. "Easy & Quick": daily use of neutron as the first step of characterization
   by in-house compact neutron source

R &D of new structural materials

TEM is mighty tool !

new alloys

composition 1-process A | comp 2-process A
comp.1-process B | comp.2-process B

mechanical grinding

electrochemical or ion milling or FIB

thin film for TEM observations

composition 1-process A | comp 2-process A
comp.1-process B | comp.2-process B

TEM by researcher・experienced technician

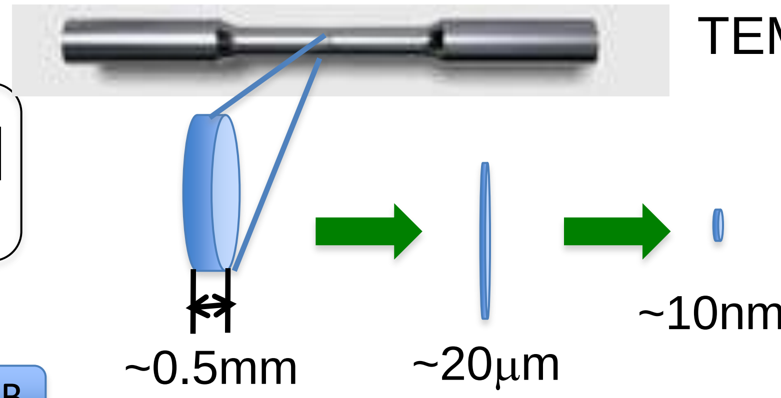
qualitative observation

composition 1-process A | comp 2-process A
comp.1-process B | comp.2-process B

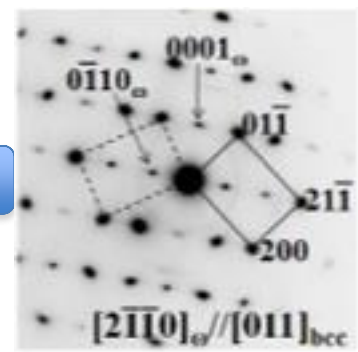
statistical analysis

quantitative information

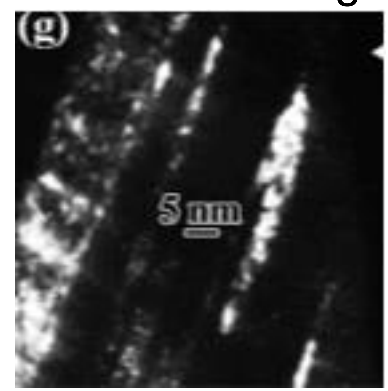
composition 1-process A | comp 2-process A
comp.1-process B | comp.2-process B



diffraction pattern

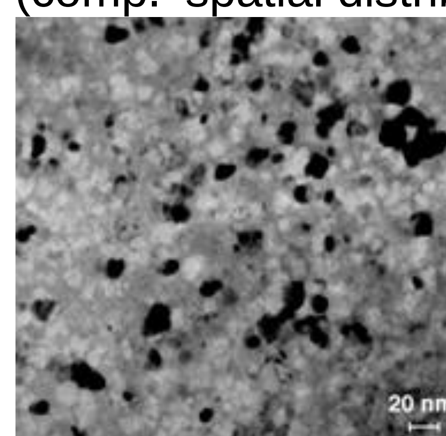


dark field image



local information (comp.・spatial distribution)

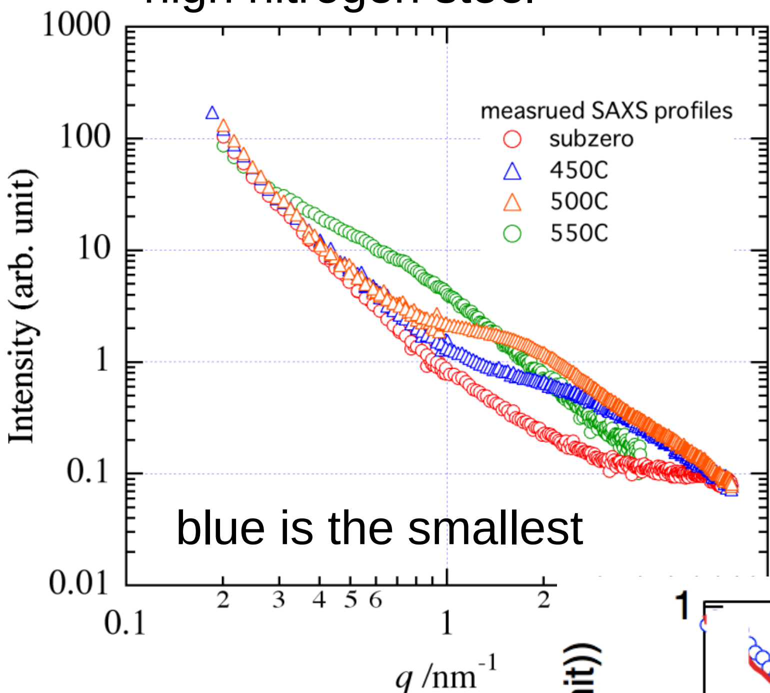
another analysis for statistic representative information



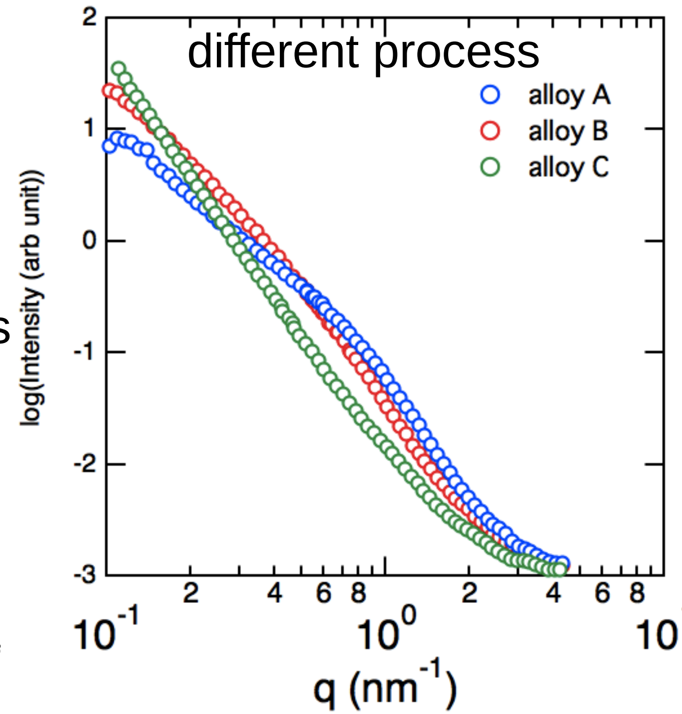
easier and faster techniques are required

by SAS easy to find which is the smallest, or which are same !

high nitrogen steel

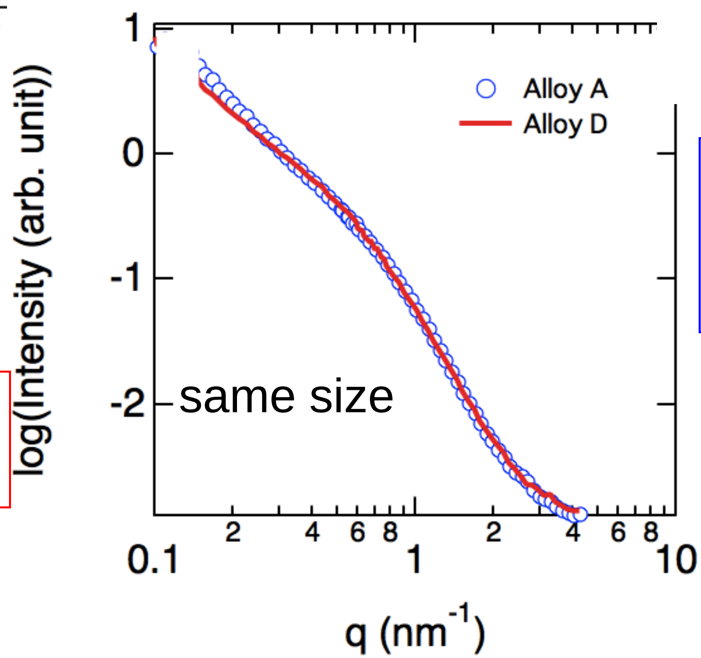


example of steels



same hardness but different comp

usable for quality control or failure analysis



all are by SAXS
→need mechanical grinding
down to~20μm for steel

for easy use, high penetration is required
→ Neutron

R &D of new structural materials

new alloys
- composition 1-process A
- comp 2-process A
- comp.1-process B
- comp.2-process B

mechanical grinding

electrochemical or ion milling or FIB

thin film for TEM observations
- composition 1-process A
- comp 2-process A
- comp.1-process B
- comp.2-process B

TEM by researcher・experienced technician

qualitative observation
- composition 1-process A
- comp 2-process A
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statistical analysis

quantitative information
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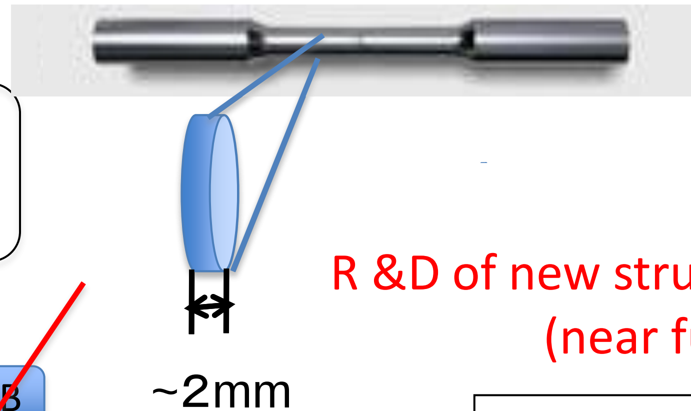


R &D of new structural materials (near future)

new alloys
- composition 1-process A
- comp 2-process A
- comp.1-process B
- comp.2-process B

small angle neutron scattering → quantitative info.

shortening time by screening samples

mechanical grinding

electrochemical or ion milling or FIB

thin film for TEM observations
- comp 1-process A
- comp.2-process B

TEM by researcher・experienced technician

detailed analysis by TEM
- comp 1-process A
- comp.2-process B

Innovative Structural Materials Association, ISMA

starting from 2013

president T. Kishi

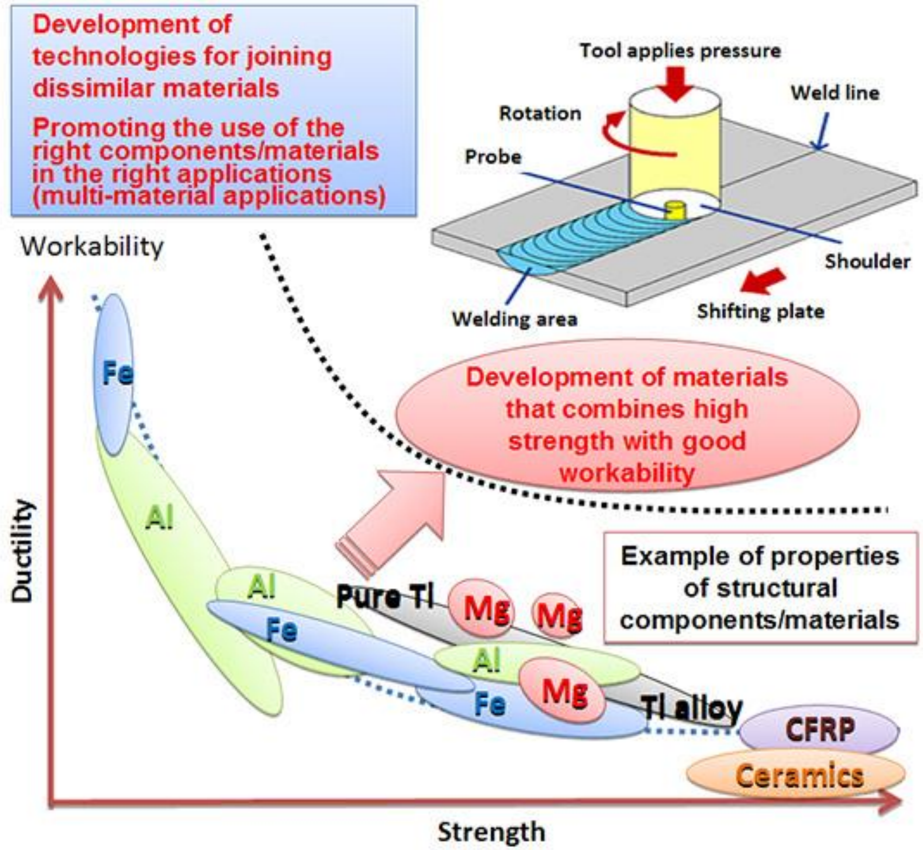
(Prof. Emeritus of Tokyo Univ.)

36 companies from Industry,

3.8 billion JPY (~ 35 million $) /year

- Steels (~ 0.5 billion JPY)
- Aluminum
- Titanium and Magnesium alloys
- CFRP
- Welding and Joint

:http://isma.jp/index.html



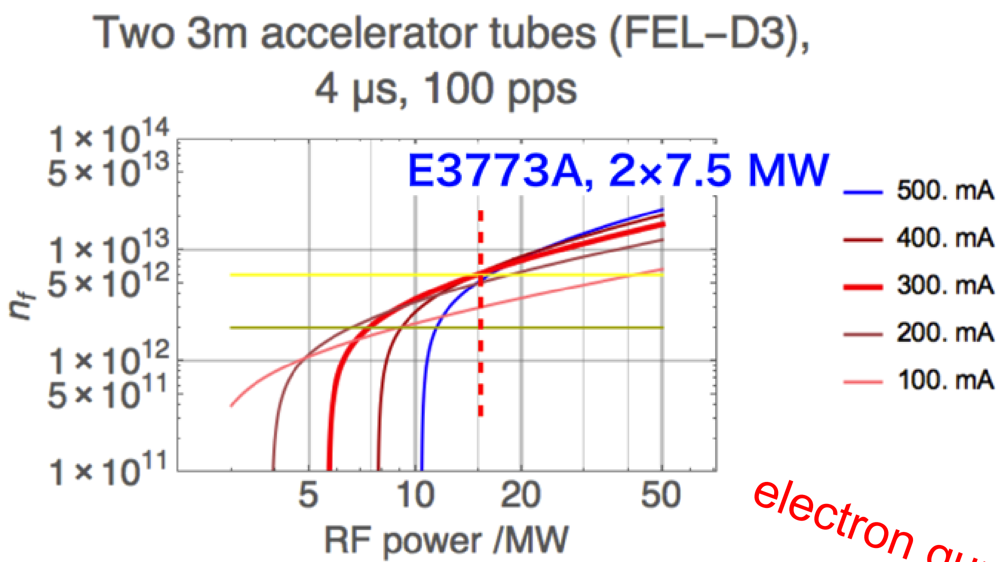
objective

For the drastic reduction of weight (by half) of transportation equipment, primarily automobiles, ISMA promotes the development of innovative materials joining technologies required to properly use newly developed materials in the right application, combined with the development of technologies involved in enhancement of the strength of major structural materials, such as iron and steel, non-ferrous, carbon-fiber-reinforced plastic (CFRP) materials, for transport equipment.

Replacing our Accelerator is under way, ~

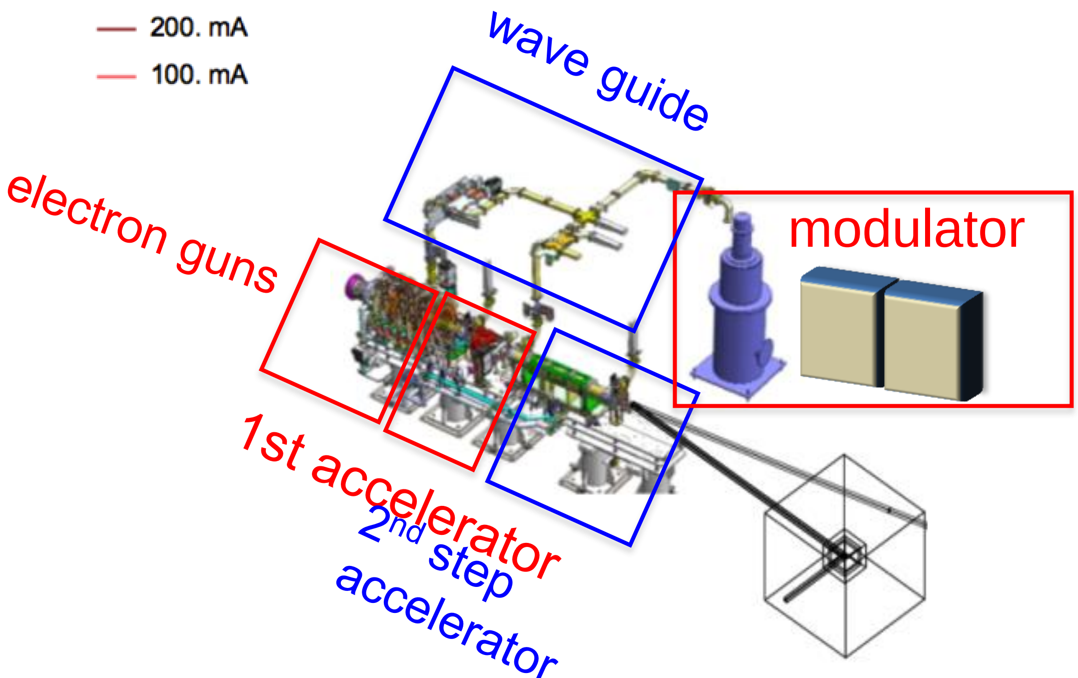
JPY 400,000,000
DKK 20,000,000

HUNS II → Neutron flux will be 3 times larger than HUNS I (from 2018)



Half of them (red) have been prepared

Remains (blue) will be

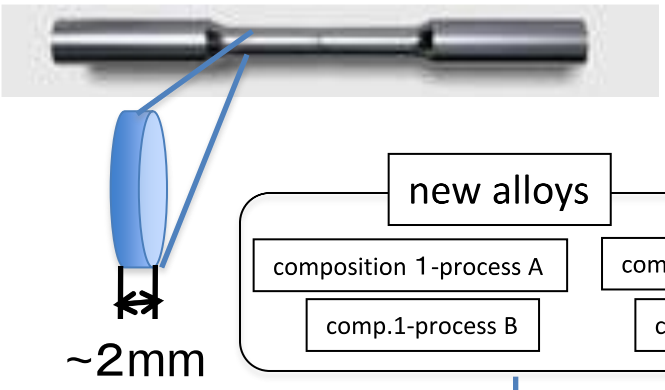


higher fiux of
neutron ~3 times

Remarks: CANS itself is a tool for frontier research in materials Science

backstage tool for accelerating materials development

embedded nanostructure analysis by SAXS and SANS



metastable phase with 1~2nm

new sight in metallurgy

new alloys

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small angle neutron scattering

quantitative info.

shortening time by screening samples

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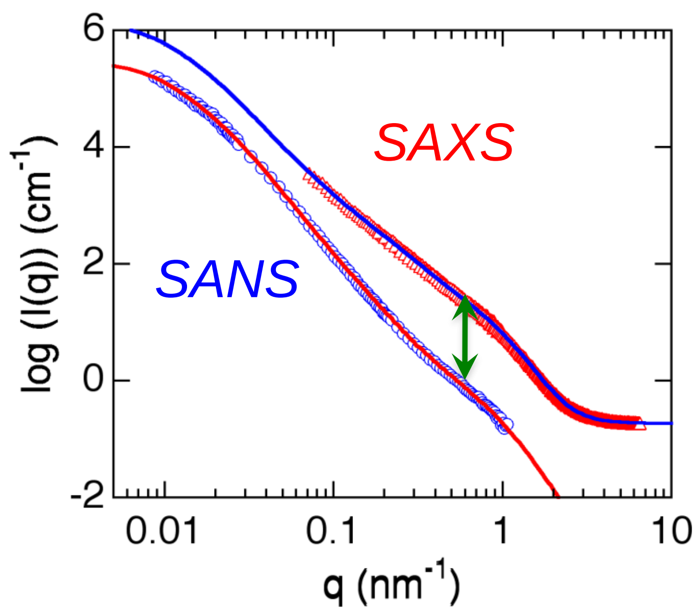
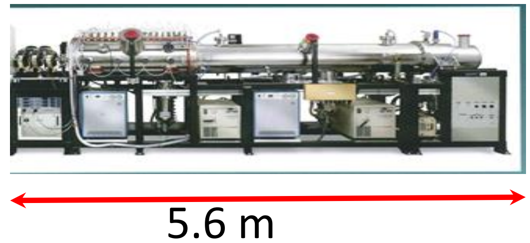
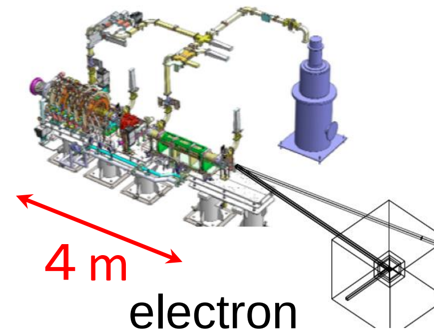


Thank you for your attention !

Accelerator driven neutron source in JAPAN

Comparison of three different neutron source in Japan

JPY 400,000,000
DKK 20,000,000

	J-PARC	Riken	2nd generation of HUNS
			
accelerated	proton	proton	electron
pulse width	0.7 μs	100 μs	3 μs
neutron	48 μs	not clear	48 μs
power	300kW(1 MW)	0.7 kW	2-3 kW
creation	spallation	P(Be, n)	(e, γ)(γ, n)
flux	10^8 n/cm^2/s	10^5 n/cm^2/s	10^5 n/cm^2/s
feature	world top 3	carrierable	stability, easy controll
	time revolution	high efficiency	short pulse
weak point	competitive	stability of beam	heavy sielding
best for	all instruments	radiography	SANS, Bragg-edge
		possibly SANS	possibly diffraction
terms	3~6 months	anytime	anytime
best match in	frontier science	civil engineering	materials science