PNC T ~~S~~J230 78-02 Tr

Thermal Performance Test on Serpentine Concrete for Fast Breeder Reactor Shielding (III)

April, 1979

POWER REACTOR AND NUCLEAR FUEL DEVELOPMENT CORPORATION

Nuclear Fuel Development Corporation.

Not For Publication
PNC TJ230 78-02 Tr
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Thermal Performance Test on Serpentine Concrete for Fast Breeder Reactor Shielding (III)

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Abstract

This report describes the results of the measurement on the thermal properties of two kinds of serpentines and the chemical analysis of serpentines and cement, which was subsequently carried out after the thermal performance test on serpentines concrete for Fast Breeder Reactor Shielding. Two kinds of serpentines used for this test were Miyamori product in Iwate prefecture and Ube product in Yamaguchi Prefecture.

The binder of concrete was normal portland cement. Unit cement weight was 400 kg/m^3 and water to cement ratio ranged from 0.50 to 0.52 and sand-ratio was 0.53.

The aggregate crushing value according to British Standard Institution (B.S. 812-1975) was tested after heating at 90°C, 110°C, 300°C and 600°C. The concrete test pieces were exposed to high temperatures of 110°C, 300°C and 600°C for 7 days and with the heating rate of 50°C per hour.

The fundamental results obtained throughout the test are as follows.

(1) All the serpentines contained less uranium and thorium than cement did.

(2) Aggregate crushing value before heating were different

among the products. Aggregate crushing value fairly increased after heating at 110°C ∿ 300°C. Aggregate crushing value markedly decreased after the heating at more than 600°C.

(3) The mechanical properties of serpentines concrete before heating showed no difference among the products. The properties of those concrete after heating at 110°C, 300°C and 600°C showed product-dependent differences. After 300°C heating, in the case of the concrete using unsound aggregate, the strength slightly decreased, while changes of length and weight were large and in the case of concrete having much crystalline water in serpentine showed little decrease of the dynamic modulus of elasticity. While after 600°C heating, mechanical properties of all the concrete were outstandingly deteriorated. When the concrete test pieces are left in the atmosphere for long time after heating, all the concrete test pieces exposed to the temperature of 300°C did not collapse, but those exposed to 600°C collapsed into smaller fragments.

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* The work performed under the contracts between Power Reactor and Nuclear Fuel Development Corporations and Electric Power Development Co., Ltd.

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APPENDIX TABLE 1 AN ESTIMATED AND PRODUCED AMOUNTS OF SERPENTINES USED FOR TEST

1. PREFACE

The authors have been experimenting successively on the properties of serpentines which are effective for shielding neutrons of the Fast Breeder Reactor and on the heat-resistant characteristics of serpentines concrete. The experiments were investigation of serpentines produced in various regions, comparison test of heat-resistance between serpentines and sandstone concrete, long term high temperature heating test of serpentines concrete combined by various kind of cement, and investigation of the effects of long term cyclic heating, heating rate, moisture content, super high temperature, etc. on serpentines concrete.

These experimental research gave us comprehension of fundamental properties of serpentines produced at several areas in Japan and heat-resistant characteristic of serpentines concrete produced at Ogose, Saitama prefecture which is supposed to be a typical one in Japan.

The present experiment is to investigate an influence of the locality of the aggregates on heat-resistant characteristic of concrete. Two kinds of serpentines were selected and anti-thermal crushing resistance of aggregates and heat-resistant characteristics of the concretes composed of these materials were examined comparing with the test results of Ogose product already given. Cement and serpentines used were chemically analyzed and also checked whether they contained elements of fissile materials or not.

2. MATERIALS USED FOR TEST

(1) Cement

In the present research the normal portland cement, manufactured by Saitama works of NIPPON Cement Co., Ltd. same with that used by the previous research was used to make a comparison under the equal condition. Chemical and physical properties of the cement is shown on item I of the tables 1 and 2. Items I and II show properties of that used in the previous research.[1)2)]

The normal portland cement was used simply because it showed more favorable result than others in the previous research.[3)]

Table 1 Chemical components of the cement in % (Unit: %)

Items	Ignition loss (ig loss)	Insoluble (in sol)	Silicon dioxide (SiO_2)	Aluminum oxide (Al_2O_3)	Iron oxide (III) (Fe_2O_3)	Calcium oxide (CaO)	Magnesium oxide (MgO)	Sulphur trioxide (SO_3)	Total
I	0.4	0.2	22.2	5.2	3.2	64.7	1.2	2.1	99.20
II	0.6	0.3	22.2	5.0	3.0	64.2	1.3	2.4	98.73
III	0.6	0.2	22.0	5.4	3.0	64.1	1.1	2.1	98.60

Table 2 Physical properties of the cement

Items	Specific gravity	Extent of powder (grains specific surface area (cm^2/g)	Condensation					Stability (pat-soaking method)	Flow value (mm)
			Room temperature (°C)	Humidity (%)	Amount of water (%)	Starting time (hour-minute)	Finished time (hour-minute)		
I	3.16	3,160	20.0	91	28.0	2-32	3-57	Good	250
II	3.15	3,090	19.0	90	27.5	2-29	4-07	Good	248
III	3.15	3,350	20.9	96	26.5	3-00	4-05	Good	247

Items	Bending strength (kg/cm^2)			Compressive strength (kg/cm^2)		
	3 days	7 days	28 days	3 days	7 days	28 days
I	31	44	66	139	231	402
II	32.9	47.5	74.1	135	222	404
III	30.7	45.0	66.7	127	216	401

(2) Aggregates

Serpentine produced at Miyamori in Iwate prefecture and at Ube in Yamaguchi prefecture were selected as aggregates for concrete considering the content of crystalline water, the locality, and quality as concrete which were revealed through the previous test.[4] Raw material to be used were crushed into gravel and sand by jaw-crusher and rodmill.

Physical properties of serpentines aggregates are shown on the table 3 together with those of Ogose product previously used.

Table 3 Physical properties of aggregates

Locality	Coarse aggregates				Fine aggregates			Ignition loss (%)
	Specific gravity	Amount of absorbed water (%)	Stability (%)	Wear loss (%)	Specific gravity	Amount of absorbed water (%)	Stability (%)	
Miyamori	2.61	2.05	6.8	13.4	2.61	2.05	7.5	8.2
Ube	2.55	2.64	13.4	20.1	2.54	2.00	13.4	14.3
Ogose (I)	2.62	1.14	7.0	14.2	2.62	1.39	6.2	16.0
Ogose (II)	2.63	1.14	8.0	15.8	2.61	1.16	6.6	15.3

Ogose and Ube products contains more crystalline water than Miyamori product judging from heat loss (110 - 800°C). Miyamori and Ogose products are tough and satisfactory as an aggregate for concrete considering physical properties, while Ube product is loose and seems unfavorable. The result of sieve analysis of aggregates used for concrete is normal as shown on the table 4.

Table 4 Sieve analysis of aggregates used for concrete

(Unit: %)

Locality	Coarse aggregates		Fine aggregates						
	20∿10 (mm)	10∿5 (mm)	5∿2.5 (mm)	2.5∿1.2 (mm)	1.2∿0.6 (mm)	0.6∿0.3 (mm)	0.3∿0.15 (mm)	Pon	Finness modulus
Miyamori	60	40	11	23	27	19	12	8	2.77
Ube	60	40	10	27	27	17	11	8	2.71
Ogose(I)	60	40	10	23	28	17	12	10	2.72
Ogose(II)	60	40	6	22	29	19	14	10	2.57

Serpentines used for chemical analysis is not exactly the same as those employed for concrete in their physical properties because they were sampled from the mine to indicate the specific property of the rock bed on the whole.

3. TEST ITEMS AND METHODS

(1) Chemical Analysis of Cement and Serpentines and Test Methods

The analysis test was entrusted to Nippon Consultant K.K. Items and methods of the chemical analysis are shown on the tables 5 and 6. Minute components were also analyzed as well as general components. Serpentines were ground into fine powders with an earthen ware mortal or something like that before analyzing.

Table 5 Method of analysis of cement

Classification	Items analized	Method of analysis
General analysis	1. Ignition loss igloss	JIS R 5202 Chemical analysis method of portland cement
	2. Insoluable insol	"
	3. Silicon dioxide SiO_2	"
	4. Aluminum oxide Al_2O_3	"
	5. Iron oxide (II) FeO	JIS M 8213 Method of quantitative analysis of iron oxide (II) in iron ore
	6. Iron oxide (II) Fe_2O_3	JIS R 5202 Chemical analysis method of portland cement
	7. Calcium oxide CaO	"
	8. Magnecium oxide MgO	"
	9. Sulphur dioxide SO_2	JIS R 5202 6.10 Method applied accordingly to that of quantative analysis of sulphates
	10. Sulphur trioxide SO_3	JIS R 5202 Chemical analysis method of portland cement

Classification	Items analized		Method of analysis
Minute analysis	11. Sodium oxide	Na_2O	JIS R 5202 Chemical analysis method of portland cement
	12. Kalium oxide	K_2O	"
	13. Absorbed water	$H_2O^{(-)}$	Weight loss when test materials are dried at 110°C
	14. Lead	Pb	Standard test method of the cement association CA JSI-51 The quantitative analysis of minute components in cement and cement's raw material.
	15. Zinc	Zn	"
	16. Cupper	Cu	"
	17. Antimony	As	"
	18. Manganese	Mn	Standard test method of the cement association CA JSI-43 The quantitative analysis of manganese in cement and dust.
	19. Cobalt	Co	" The quantitative analysis according to CAJSI-51
	20. Cadmium	Cd	" The quantitative analysis of minute components in cement and cement's raw material.
	21. Chlorine	Cl	The quantitative analysis of chlorine in hardened concrete by chemical specialist committee of the cement association
	22. Titanium oxide	TiO_2	The quantitative analysis by the absorption intensity of hydrogen perioxide
	23. Nickel oxide	NiO	Quantitatively analyzed according to the standard test method CAJSI-51 by the cement association
	24. Chromium	Cr	The standard test method of the cement association CAJSI-51 The quantitative analysis of minutes components in cement and cement's raw material.

Classi-fication	Items analized		Method of analysis
Minute analysis	25. Chromium ion	Cr^{6+}	The standard test method of the cement association CAJSI-51 The quantitative analysis of minutes components in cement and cement's raw material.
	26. Fluorine	F	The quantitative analysis of fluorine with water-heat decomposition/ion meter, direct analysis, by Nippon Cement K.K.
	27. Total mercury	T-Hg	The standard test method of the cement association CAJSI-48 The quantitative analysis of mercury in cement and dust
	28. Phosphorus	P	Converted method of the quantitative analysis of phosphorus pentadoxide according to the chemical analysis of limestone JIS M8850
	29. Uranium	U	Method of the National Geology Research
	30. Zirconium	Zr	The absorption intensity method with quercetin
	31. Thorium	Th	Method of the National Geology Research
	32. Boron	B	Converted quantitative analysis of B_2C_3 from Manit method, ASTM C 169-75

Table 6 Methods of analysis of serpentines

Classification	Items analyzed	Methods of analysis
General analysis	1. Ignition loss igloss	The standard test method of the cement association CAJSI-12 The chemical analysis of siliceous raw materials
	2. Silicon dioxide SiO_2	"
	3. Aluminum oxide Al_2O_3	"
	4. Iron oxide (II) FeO	JIS M 8213 The quantitative analysis of iron oxide (II) in iron ore.
	5. Iron oxide(III) Fe_2O_3	The standard test method of the cement association CAJSI-12 The chemical analysis of siliceous raw materials.
	6. Calcium oxide CaO	"
	7. Magnecium oxide MgO	"
	8. Sulphur dioxide SO_2	JIS R 5202 6.10 Method according to the quantitative analysis of sulphate sulphur
	9. Sulphur trioxide SO_3	The quantitative analysis of sulphur trioxide by the quantitative method of sulphate salt in a test sample of siliceous salt
	10. Sodium oxide Na_2O	The standard test method of the cement association CAJSI-12 The chemical analysis of siliceous raw materials
	11. Kalium oxide K_2O	"
	12. Absorbed water $H_2O^{(-)}$	Weight loss when test materials are dried at 110°C
	13. Crystalline water $H_2O^{(+)}$	Difference between igloss % and $H\ O^{(-)}$ %
	14. Lead Pb	The standard test method of the cement association CAJSI-51 The quantitative analysis of minute components in cement and cement's raw material

Classification	Items analyzed		Methods of analysis
Minute analysis	15. Zinc	Zn	The standard test method of the cement association CAJSI-51 The quantitative analysis of minute components in cement and cement's raw material
	16. Cupper	Cu	"
	17. Antimony	As	"
	18. Manganese	Mn	" CAJSI-51 The quantitative analysis of manganese an cement and dust
	19. Cobalt	Co	" Method applied according to CAJSI-51
	20. Cadmium	Cd	" The quantitative analysis of minute components in cement and cement's raw material.
	21. Chlorine	Cl	The quantitative analysis of chlorine in hardened concrete by chemical specialist committee of the cement association
	22. Titanium oxide	TiO_2	The quantitative analysis by the absorption intensity of hydrogen peroxide
	23. Nickel oxide	NiO	Quantitatively analyzed according to the standard test method CAJSI-51 by the cement association
	24. Chromium	Cr	The standard test method of the cement association CAJSI-51 The quantitative analysis of minute components in cement and cement's raw material
	25. Chromium ion	Cr^{6+}	"
	26. Fluorine	F	The quantitative analysis of fluorine with water-heat decomposition ion meter, direct analysis by Nippon cement K.K.
	27. Total mercury	T.Hg	The standard test method of the cement association CAJSI-48 The quantitative analysis of mercury in cement and dust

Classi-fica-tion	Items analyzed		Methods of analysis
	28. Phosphorus	P	Converted method of the quantitative analysis of phosphorus pentadoxide according to the chemical analysis of limestone JIS M 8850
	29. Uranium	U	Method of the National Geology Research
	30. Zirconium	Zr	The absorption intensity method with quercetin
	31. Thorium	Th	Method of the National Geology Research
	32. Boron	B	Converted quantitative analysis of B_2O_3

(2) Method of Anti-Thermal Crushing Test of Serpentines

This crushing test was performed according to British standard, BS 812; 40 tons load method and 10% crushing method. Test materials were filled in a cylinder of 150mm in diameter and 140mm in height up to 100mm high and loaded to crush by the plunger. The size of test materials were 10 ∿ 15 mm according to the standard.

Heating temperatures were 90, 110, 300, and 600°C. The materials were heated for 4 hours in case of 90°C (corresponds to BS 812), and for one week in case of 110 ∿ 600°C. Test materials were cooled in the atmosphere just after heating until reaching the room temperature. Test materials heated at the temperature of 600°C were also left in the atmosphere for a week after heating.

(3) Anti, Thermal Test of Concrete and Their Methods

a) Properties of concrete used for test

Mixing of concrete were as follows same as that of the previous test.[1][2] Amount of water was experimentally determined so as to give a predetermined slump.

Mixing of concretes

Max. size of aggregates	Weight of cement per unit	Percentage of fine aggregates	Range of slump
20 mm	200 Kg	53%	5 ∿ 10 cm

Concretes were mixed by the single drum mixer of 280ℓ in volume according to the following process, two minute's mixing → one minute holding → one minute's mixing. Proportion of fresh concrete and the properties are shown on the tables 7 and 8, showing almost same values of W/C as that of Ogose product.

Table 7 Proportion of concrete

Kinds of concrete	Max. size of aggregates (mm)	Amount of cement C (Kg/m^3)	Amount of water W (Kg/m^3)	Absolute percentage of fine aggregates s/a (%)	Amount of fine aggregares S (Kg/m^3)	Amount of coarse aggregates G (Kg/m^3)
Miyamori (1)	20	400	204	53	901	799
Miyamori (2)	20	400	204	53	898	799
Ube (1)	20	400	202	53	876	780
Ube (2)	20	400	200	53	879	783
Ogose (1)	20	400	201	53	882	782
Ogose (2)	20	400	208	53	896	794

Table 8 Properties of concrete

Kind of concrete	Ratio of water to cement W/C (%)	Ratio of cement to vacancy (C/V)	Properties of concrete			Heating temperature
			Slump (cm)	Amount of air (%)	Temperature (°C)	
Miyamori (1)	0.51	0.51	7.7	2.0	8.0	300°C
Miyamori (2)	0.51	0.51	7.9	1.9	9.0	600°C
Ube (1)	0.51	0.51	7.6	2.4	11.0	300°C
Ube (2)	0.50	0.50	7.7	2.8	9.0	600°C
Ogose (1)	0.50	0.50	7.7	2.5	8.0	300°C
Ogose (2)	0.52	0.52	6.4	1.7	23.0	600°C

Each test sample was formed by placing concrete in a given mold being compacted with a bar vibrator, after curing for 24 hours in the atmosphere of 21 ± 3°C, removed.

b) Methods of curing and heat drying of concrete

The process of curing and heat drying of concrete are seen from Fig. 1. Concrete was cured in the fog at temperature of 21 ± 3°C for first 14 days after removing, and then in the atmosphere for next 84 days before predrying, or for next 91 days before preheating at temperature of 21 ± 3°C and humidity of 80 ± 5%. Concretes cured in the atmosphere for 84 days were predried for next one week at temperature of 110°C. Heating was performed at temperature of 300 and 600°C for one week.

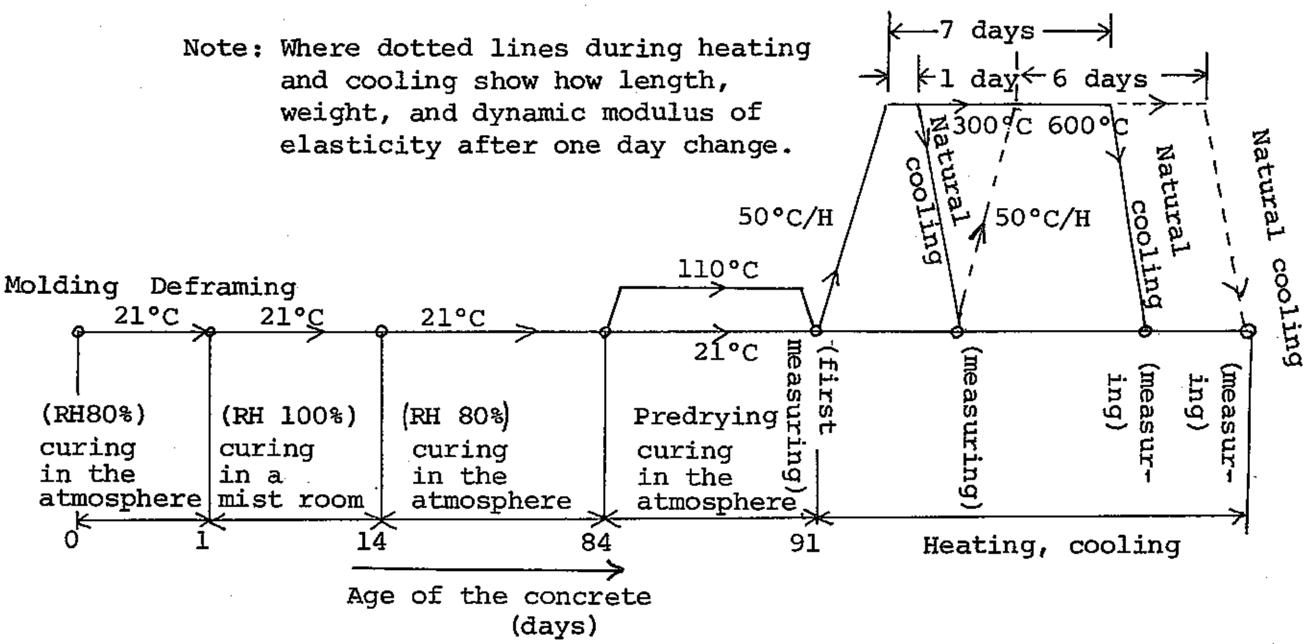

Fig. 1 The process of curing and heating

The test material was heated up at the rate of 50°/h in the beginning of heating, and measured physical properties at room temperature after cooling down in the atmosphere. The specification of the furnace is as shown on the table 9. Local temperature deviation is ±10°C at temperature of 700°C, which can be regarded almost uniform. Test materials were arranged at a sufficient interval in the furnace so that heat atmosphere could circulate uniformly.

Table 9 Outline of the drying furnace

Specs.	Drying furnace, 110°C	Drying furnace, 300°C and 600°C
Dimensions	width 105 x length 100 x height 106cm	width 165 x length 185 x height 165cm
Temperature	200°C	700°C
Capacity of heaters	4.5KW	150KW (75KW x 2)
Temperature balancing method	air circulation	air circulation
Amount of loading (ø10 x 20cm)	288 (72 x 4 layers)	252 (84 x 3 layers)
Amount of loading (ø15 x 30cm)	96 (24 x 4 layers)	144 (48 x 3 layers)

c) Heat-Resistance Tests of Concrete and Their Methods

Physical properties and test methods are shown on the table 10. Five test items required to judge the quality of the concrete were selected. Two kinds of test pieces having been given different curing condition were tested for each item; one was cured by the standard method, and another predried (refer to Fig. 1). Period of heating was decided to be 7 days considering the result of long term heat test[3]) previously performed. Test methods were complied with JIS except measurement of weight.

Table 10 Properties tested and methods

Properties tested	Life of material (days)		Test methods	Dimensions of samples (cm)
	Before heating	After heating		
Compressive strength	7,28,91	1, 7	JIS A 1108	ø10 x 20
Tensile strength	7,28,91	1, 7	JIS A 1113	ø10 x 20
Dynamic modulus of elasticity	7,28,84,91	1, 7	JIS A 1127 (Bending method)	10 x 10 x 40
Elongation	7,28,84,91	1, 7	JIS A 1124 (Contact method)	10 x 10 x 40
Change of weight	7,28,84,91	1, 7	Precision platform scale (in gram)	10 x 10 x 40

4. RESULTS OF TESTS

The test results of chemical analysis of cement and serpentines, anti-thermal crushing property of serpentines, and heat-resistance properties of concrete are described as follows.

(1) Chemical Analysis of Cement and Serpentines

Table 11 Chemical analysis of cement and serpentines

Classification		Analyzed		Unit	Normal portland cement	Serpentines	
					Product of Saitama works, Nippon cement K.K.	Ogose product in Saitama Prefecture	Miyamori product in Iwate prefecture
General analysis	1	Ignition loss	igloss	%	1.0	13.9	12.8
	2	Insoluable	insol	%	0.3	-	-
	3	Silicon dioxide	SiO_2	%	22.2	38.7	39.8
	4	Aluminium oxide	Al_2O_3	%	4.4	2.1	1.3
	5	Iron oxide (II)	FeO	%	0.04	4.2	2.1
	6	Iron oxide (III)	Fe_2O_3	%	3.1	3.2	5.4
	7	Calcium oxide	CaO	%	64.3	4.2	0.8
	8	Magnecium oxide	MgO	%	1.3	32.7	36.7
	9	Sulphur dioxide	SO_2	%	0.00	0.00	0.00
	10	Sulphur trioxide	SO_3	%	2.00	0.1	0.1
	11	Sodium oxide	Na_2O	%	0.37	0.15	0.15
	12	Kalium oxide	K_2O	%	0.60	0.02	0.08
	13	Absorped water	$H_2O^{(-)}$	%	(0.24)	(1.25)	(1.41)
	14	Crystalline water	$H_2O^{(+)}$	%	-	(12.6)	(11.4)

Classification		Analyzed		Unit	Normal portland cement	Serpentines	
					Product of Saitama works, Nippon cement K.K.	Ogose product in Saitama prefecture	Miyamori product in Iwate prefecture
Minute analysis	15	Lead	Pb	PPM	24.6	0.0	0.0
	16	Zinc	Zn	PPM	298.	60.3	59.0
	17	Copper	Cu	PPM	147.	0.0	0.0
	18	Antimony	As	PPM	19.3	0.8	3.4
	19	Manganese	Mn	PPM	204.	750.	987.
	20	Cobalt	Co	PPM	0.8	91.4	101.
	21	Cadmium	Cd	PPM	0.8	0.2	0.1
	22	Chlorine	Cl	%	0.01	0.01	0.01
	23	Titanium oxide	TiO	%	0.22	0.27	0.10
	24	Nickel oxide	NiO	PPM	15.9	2300.	2400.
	25	Chromium	Cr	PPM	59.3	2260.	2280.
	26	Chromium ion	Cr^{6+}	PPM	8.2	0.1	0.1
	27	Fluorine	F	PPM	270.	300.	130.
	28	Total mercury	T.Hg	PPB	15.	79.	37.
	29	Phosphorus	P	%	0.05	0.02	0.01
	30	Uranium	U	PPM	1.6	0.1	0.2
	31	Zirconium	Zr	PPM	80.	25.	41.
	32	Thorium	Th	PPM	2.	1.	0.
	33	Boron	B	%	0.00	0.00	0.00
		Total			100.00	100.15	99.95

Note: 0.001% = 1 PPM = 1,000 PPB

Total alkali in cement = Na_2O + 0.658 K_2O = 0.76%

Chemical analysis of the normal portland cement and serpentines (Ogose and Miyamori products) are shown on the table 11. Analysis of Uranium and Thorium was performed according to the method developed by the Geology Research of the Industrial Technology Institute using the standard reagent U_3O_8 and Th of 97.5% and 91.0% in purity respectively. Error was regarded to be within 15% of standard deviation.

a) Cement

1.6 ppm of Uranium and 2 ppm of Thorium were detected. These are elements relating to nuclear fission.

b) Serpentines

0.1 ∿ 0.2 ppm of Uranium and 0 ∿ 1 ppm of Thorium were detected as FP elements. The values have no relation with the locality and are very small compared with content in cement. It is characteristic that serpentines contain Magnecium oxide MgO by 32.7 ∿ 36.7% and crystalline water $H_2O^{(+)}$ by 11.4 ∿ 12.6% as general components.

(2) Anti-Thermal Crushing Property of Serpentines

a) Appearance

Color of each serpentine does not change below 300°C as seen on the photo 1 and shows greenish pale black, but turns yellowish brown at 600°C. No crack developed by heating.

b) Crushing resistance

Crushing values of each serpentine are shown on the table 12, and depend largely on its locality. Variations of the crushing values due to heating are similar in all products to each other seen from the 10% crushing load as shown on the Fig. No. 2. They increase as temperature increases up to 300°C, while decrease outstandingly at 600°C. The range of values is 74 ∿ 104%. A few of difference of the tendencies could be observed among the material origin.

Table 12 Crushing value of each serpentine before and after heating

Temperature (°C)	Miyamori Product		Ube Product		Ogose Product	
	Crushing value (%)	10% crushing load (t)	Crushing value (%)	10% crushing load (t)	Crushing value (%)	10% crushing load (t)
90 (BS standard)	10.7	36.4	18.7	19.8	17.6	24.7
110	10.0	40.0	17.6	23.3	12.5	31.6
300	8.0	45.0	13.0	29.8	11.5	34.0
600	19.9	19.2	29.3	9.3	20.9	16.8
600 (left alone*)	21.9	16.5	30.0	9.3	23.0	15.8

Note 1) BS standard is a method that test materials are tested after 4 hour's drying at 90°C.

Note 2) Heating period at 110 ~ 600°C was 7 days.

Note 3) Marked * was tested after it had been left alone in the atmosphere for 7 days after heating.

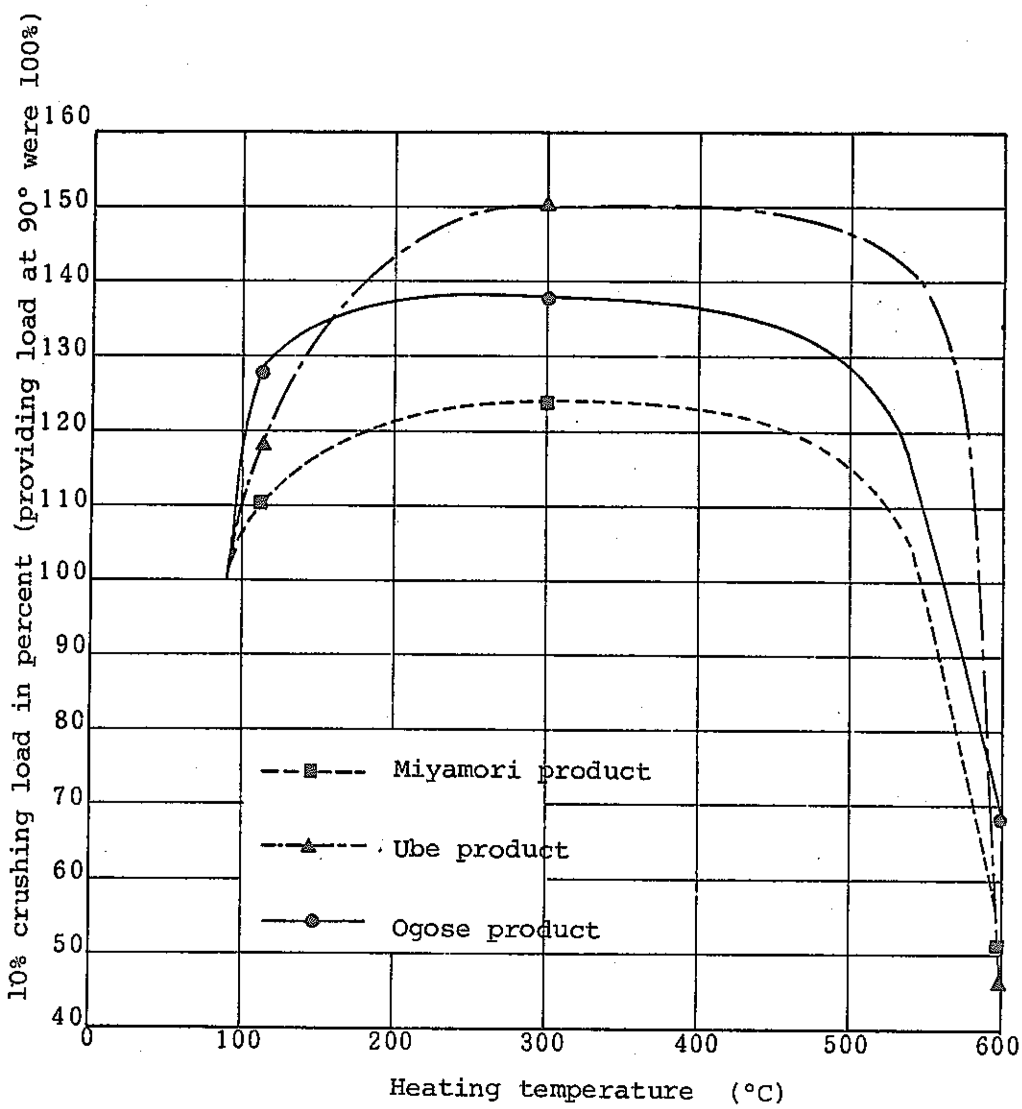

Fig. 2 Relationship between heating temperature and 10% crushing load of serpentines

(3) Heat-Resistance Properties of Concretes

a) Appearance of concrete

Photo 2 shows appearance of concrete made from each serpentine. Haircracks occurred on the surface just after the sample was taken out from the furnace and cooled rapidly in the atmosphere after heating at 300°C, but they didn't develop after leaving alone in the atmosphere for long period. When heated at 600°C, cracks occurred gradually developed through leaving alone in the atmosphere for long period and finally led to collapse of concrete.

b) Compressive strength

The test result is shown on the table 13. As long as compressive strength of concrete before heating is concerned, fluctuation depending upon products is less than that on mixing lot. Compressive strength after predrying and heating is shown on Fig. 3. Results are as follows; (1) A little increase by predrying at 110°C. (2) 300°C heating gives an increase or decrease depending upon aggregates. Predried shows relatively large decrease. (3) 600°C heating gives an extraordinary decrease in compressive strength; the residual compressive strength is 150 - 160 kg/cm^2 about one thirds of strength before heating regardless of predrying or locality. (4) Heating period more than 1 day makes little effect on compressive strength, while heating period within one day make an outstanding effect. Thus it concludes that long term heating gives no substantial influence to compressive strength.

Table 13. Compressive strength of concrete before and after heating

Kinds of concrete		Heating temperature (°C)	Curing	Compressive strength (Kg/cm^2)				
				Material life of curing (days)			Material life of heating (days)	
				7	28	91	1	7
Miyamori	(1)	300	Standard	296	443	480	468	451
			Dry	(Material life of 84∿91 days)		483	410	414
	(2)	600	Standard	279	449	467	134	158
			Dry	(Material life of 84∿91 days)		515	173	156
Ube	(1)	300	Standard	303	468	482	534	507
			Dry	(Material life of 84∿91 days)		493	464	493
	(2)	600	Standard	252	413	448	159	152
			Dry	(Material life of 84∿91 days)		493	155	152
Ogose	(1)	300	Standard	278	460	460	443	432
			Dry	(Material life of 84∿91 days)		477	403	393
	(2)	600	Standard	289	440	523	180	169
			Dry	(Material life of 84∿91 days)		483	175	160

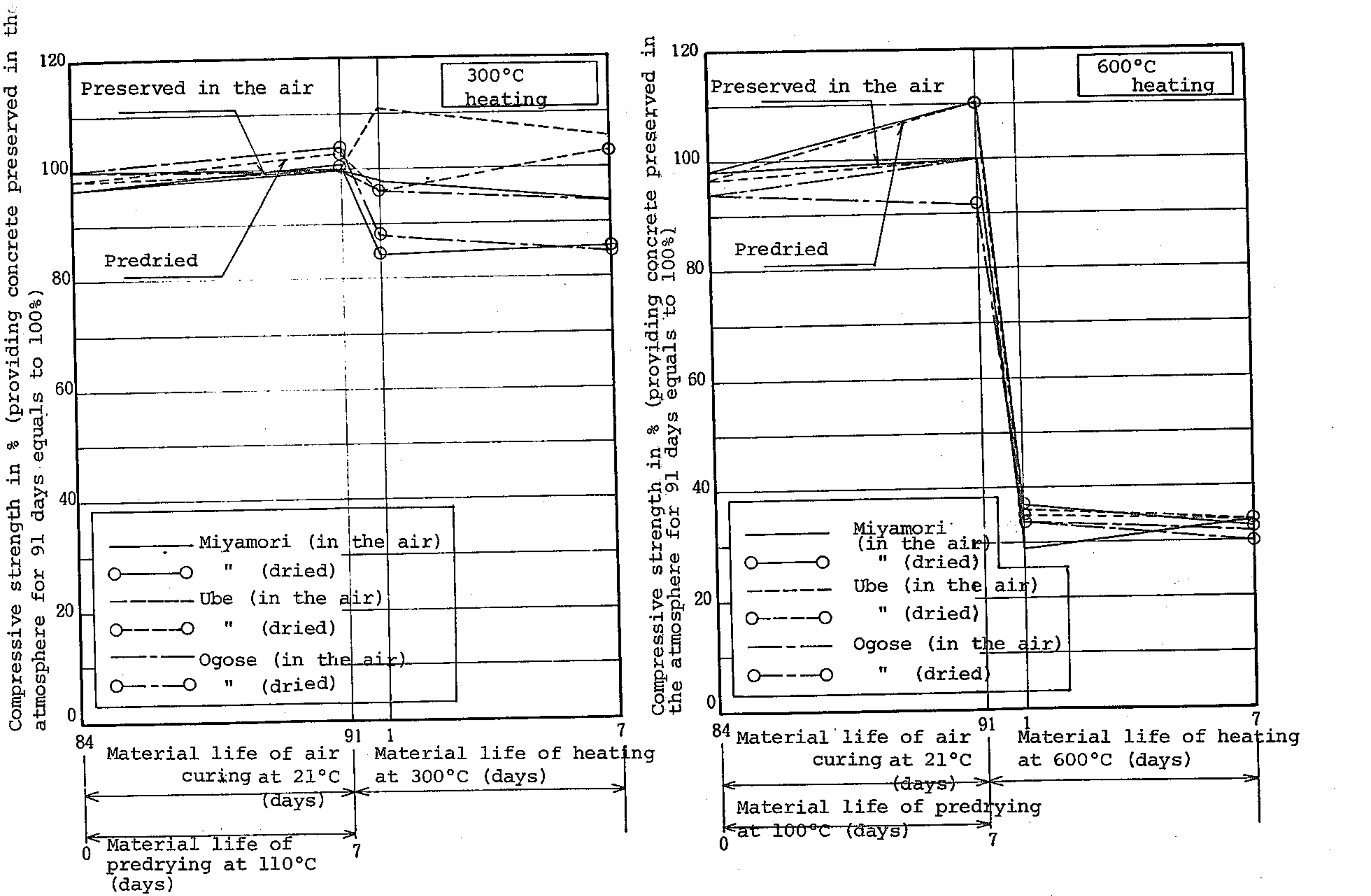

Fig. 3 Relationship between heat drying and compressive strength of concrete

c) Tensile strength

The test result is shown on the table 14. The locality of the aggregate has slight influence on tensile strength of concrete before heating. Tensile strength changes due to drying and heating as follows which is obvious from Fig. 4; (1) 5 ∿ 20% decrease by 110°C drying, which depends upon the locality of the aggregate. (2) 0 ∿ 20% decrease by 300°C heating, which also depends upon the locality of the aggregate. Predried one shows further decrease by 6 ∿ 8%. (3) By 600°C heating, the residual tensile strength decreases largely to 10 ∿ 11 kg/cm^2 regardless of predrying or the locality of the aggregate. (4) Heating period more than 1 day makes little effect on tensile strength, while heating period within one day make an outstanding effect. Thus it concludes that long term heating gives no substantial does not influence to tensile strength.

Table 14 Tensile strength of concrete before and after heating

Kind of concrete		Heating temperature (°C)	Curing	Tensile strength (Kg/cm^2) Life material of curing (days) 7	28	91	Life material of heating (days) 1	7
Miyamori	(1)	300	Standard	30	34	36	33	33
			Dry	(Material life of 84∿91 days)		32	29	31
	(2)	600	Standard	28	32	35	10	11
			Dry	(	")	28	12	11
Ube	(1)	300	Standard	27	30	36	35	36
			Dry	(	")	34	33	34
	(2)	600	Standard	23	26	31	13	11
			Dry	(	")	26	13	11
Ogose	(1)	300	Standard	28	37	39	31	31
			Dry	(	")	32	29	28
	(2)	600	Standard	27	36	42	12	11
			Dry	(	")	34	12	10

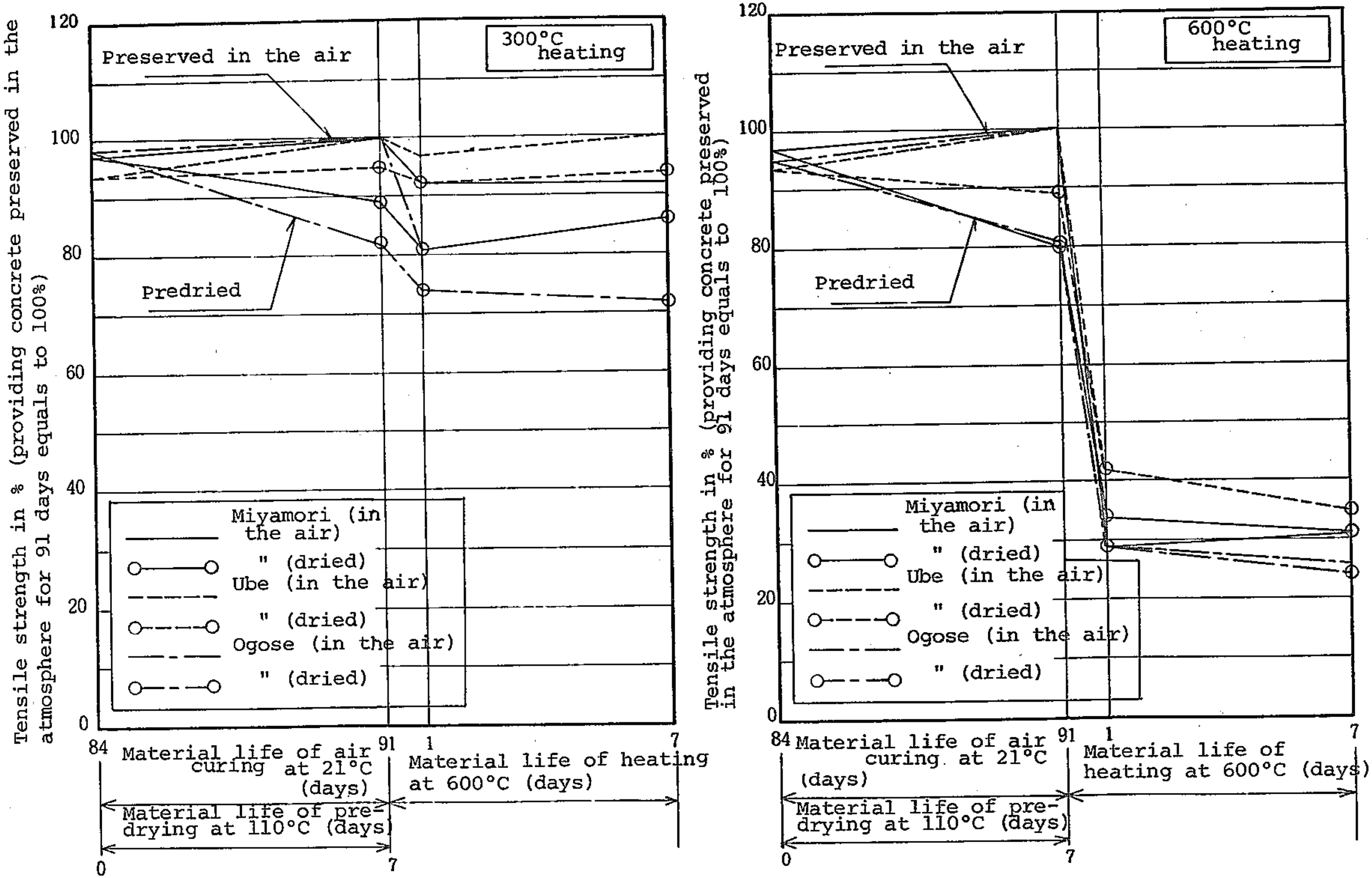

Fig. 4 Relationship between heat drying and tensile strength of concrete

d) Dynamic Modulus of Elasticity

The test result is shown on the table 15. Dynamic modulus of elasticity of concrete before heating depends slightly upon the locality of the aggregate. That after heating is as follows which is obvious from Fig. 5; (1) 12 ∿ 17% decrease by 110°C drying regardless of the locality of the aggregate. (2) 49 ∿ 61% decrease by 300°C heating, which depends upon the locality of the aggregate but not upon predrying. (3) 91 ∿ 94% decrease by 600°C heating. Effect of the locality of the aggregate and predrying is negligible. (4) Continuous heating causes a little decrease in dynamic modulus of elasticity through 1 ∿ 7 days. Thus it concludes that continuous heating have an influence on dynamic modulus of elasticity.

Table 15 Dynamic modulus of elasticity of concretes before and after heating

Kinds of concrete		Heating temperature (°C)	Curing	Dynamic modulus of elasticity				
				Material life of curing (days)			Material life of heating (days)	
				7	28	91	1	7
Miyamori	(1)	300	Standard	327	361	370	168	144
			Dry	(material life of 84 ∿ 91 days)		280	171	145
	(2)	600	Standard	328	366	369	48	25
			Dry	(")		285	49	28
Ube	(1)	300	Standard	290	313	315	160	135
			Dry	(")		240	170	147
	(2)	600	Standard	278	304	309	40	17
			Dry	(")		228	48	27
Ogose	(1)	300	Standard	322	327	327	198	178
			Dry	(")		255	193	174
	(2)	600	Standard	340	363	374	62	21
			Dry	(")		273	67	22

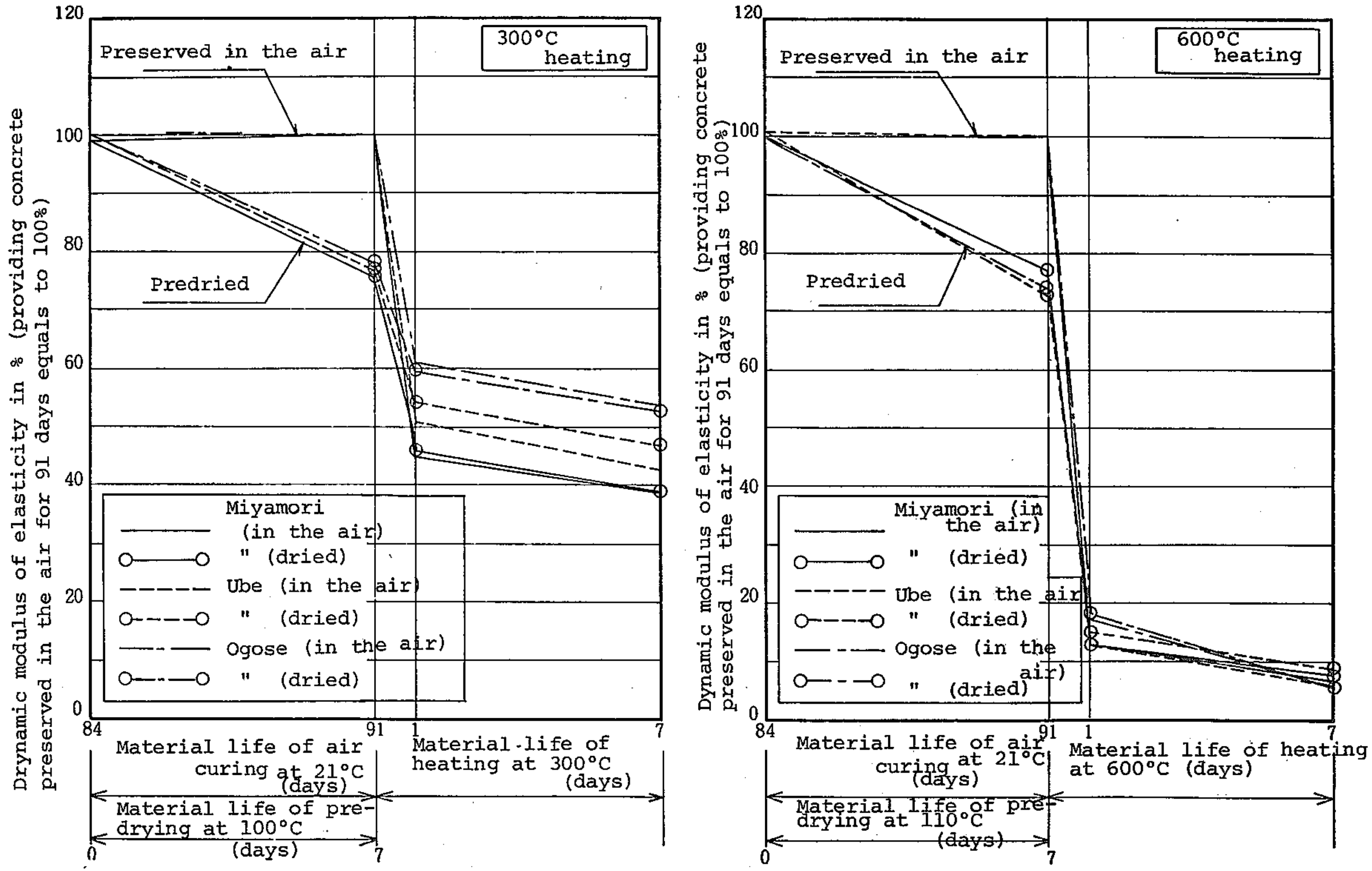

Fig. 5 Relationship between heat drying and dynamic modulus of elasticity of concrete

e) Elongation

The test result is shown on the table 16. Fluctuation of the elongation of concrete through curing period due to the locality of the aggregate, is unclear and within that of mixing lot. The elongation after heating is as follows, which is obvious from Fig. 6; (1) 0.07 ∿ 0.17% shrinking by 110°C drying. It depends largely on the locality of the aggregate. (2) 0.20 ∿ 0.45% shrinking by 300°C heating, which also depends on the locality of the aggregate. Predried one shrinks further. (3) Even 0.63 ∿ 1.61% shrinkage occurs by 600°C heating. It shows outstanding difference due to the locality of the aggregate. Shrinkage of predried concretes is further large. (4) A small increase of shrinkage is found through heating period of 1 ∿ 7 days at each temperature.

Table 16   Elongation of concrete before and after heating

Kind of concretes		Heating temperature (°C)	Curing	Elongation (%) Material life of curing (days)				Material life of heating (days)	
				7	28	84	91	1	7
Miyamori	(1)	300	Standard	0.021	0.009	0.001	0	-0.273	-0.277
			Dry	0.029	0.004	0	-0.102	-0.276	-0.297
	(2)	600	Standard	0.012	0.006	0.001	0	-0.935	-0.999
			Dry	0.011	0.005	0	-0.136	-1.081	-1.087
Ube	(1)	300	Standard	0.031	0.002	0.003	0	-0.407	-0.447
			Dry	0.029	0.000	0	-0.166	-0.467	-0.496
	(2)	600	Standard	0.017	0.008	0.001	0	-1.540	-1.612
			Dry	0.018	0.001	0	-0.162	-1.512	-1.614
Ogose	(1)	300	Standard	0.027	0.004	0.000	0	-0.176	-0.201
			Dry	0.040	0.006	0	-0.070	-0.198	-0.228
	(2)	600	Standard	0.018	0.010	0.002	0	-0.585	-0.625
			Dry	0.024	0.012	0	-0.094	-0.670	-0.716

Note) Original length of test materials is 100mm.

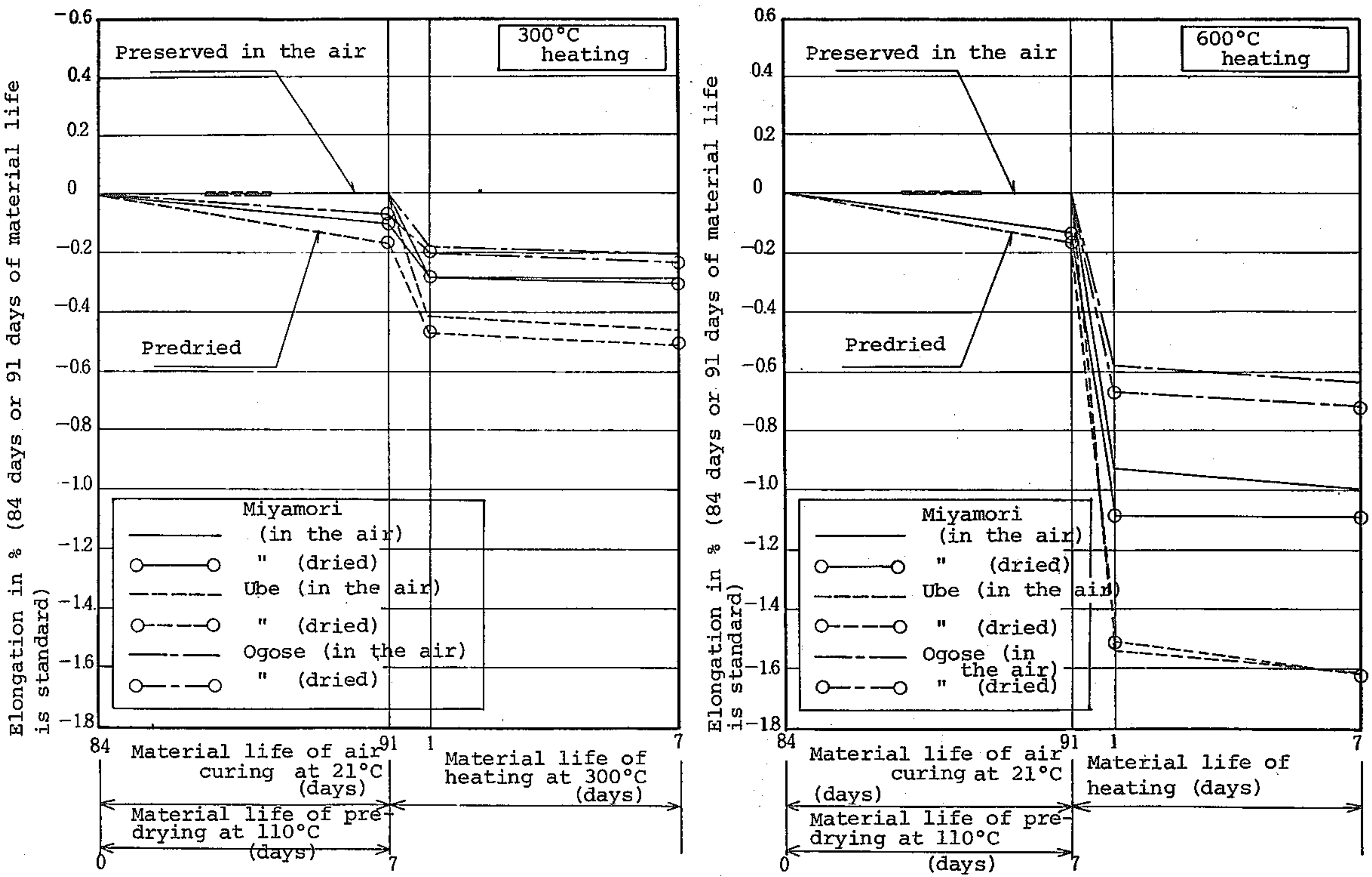

Fig. 6 Relationship between heat drying and shrinkage of concrete

f) Weight change

The test result is shown on the table 17. Weight loss of the concrete during 7 ∿ 91 days of curing is 0.2 ∿ 1.8% depending slightly upon the locality of the aggregate. Weight change after heat drying is as follows seen from Fig. 7. (1) 5.0 ∿ 6.5% loss by 110°C drying, which depends upon the locality of the aggregate. (2) 6.5 ∿ 8.5% loss by 300°C heating depending upon the locality of the aggregate but not on predrying. (3) 15.3 ∿ 18.5% loss by 600°C heating depending upon the locality of the aggregate but not on predrying. (4) Both heating period and the locality of the aggregate make a slight effect on weight change.

Table 17. Weight change of concrete before and after heating

Kind of concretes		Heating temperature (°C)	Curing	Weight change of concrete in %					
				Material life of curing in day				Material life of heating in day	
				7	28	84	91	1	7
Miyamori	(1)	300	Standard	0.45	0.23	0.03	0	-7.76	-7.84
			Dry	0.40	0.18	0	-5.87	-7.70	-7.81
	(2)	600	Standard	0.24	0.14	0.23	0	-15.14	-16.32
			Dry	0.44	0.18	0	-5.95	-15.50	-16.51
Ube	(1)	300	Standard	1.04	0.30	0.24	0	-8.33	-8.49
			Dry	0.99	0.33	0	-6.10	-8.37	-8.54
	(2)	600	Standard	0.47	0.23	0.70	0	-18.27	-18.58
			Dry	0.41	0.15	0	-6.58	-18.05	-18.56
Ogose	(1)	300	Standard	1.35	0.22	0.03	0	-6.47	-6.52
			Dry	1.31	0.22	0	-4.96	-6.47	-6.53
	(2)	600	Standard	1.70	0.90	0.00	0	-13.80	-15.30
			Dry	1.80	1.00	0	-5.00	-13.80	-15.40

Note) Sample concrete's weight at the beginning of measurement was 9.350 to 9.650 gs.

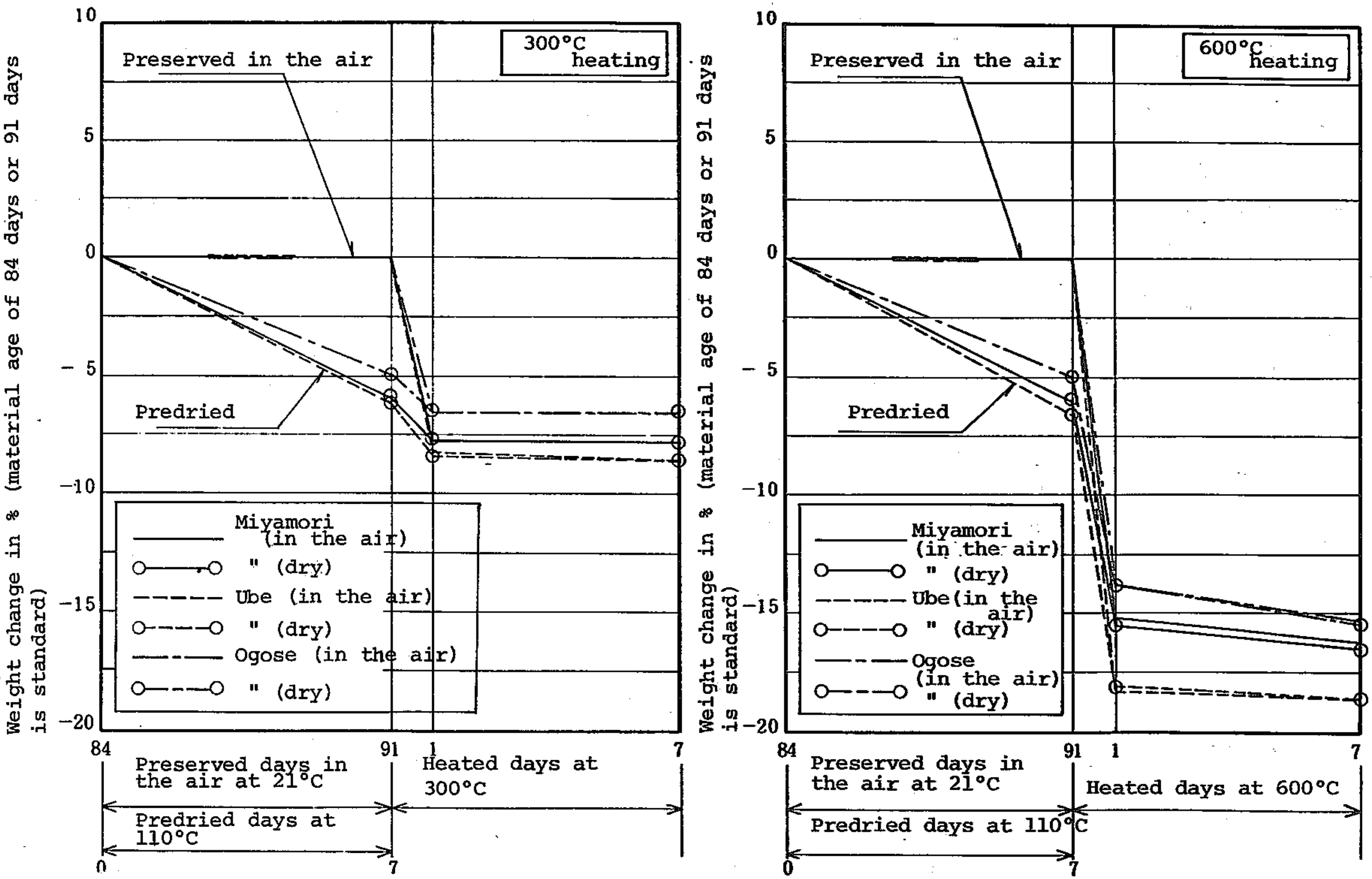

Fig. 7 Relationship between heat drying and weight change of concretes

5. DISCUSSION AND CONSIDERATION

Each item are discussed and considered below. Deterioration of the concrete was discussed by a series of research[1),2)and 3)] on heat resistance of the serpentines concrete, so that this report forcuses on the effect of the aggregate on concrete.

(1) Chemical Analysis of Cement and Serpentines

As elements relating to nuclear fission, Uranium and Thorium were detected. Their content is very small compared with the present concentration on the earth (U = 3 ∿ 4 ppm, Th = 10 ∿ 15 ppm) or content in the average granite (U = 3 ppm, Th = 17 ppm)[6)] (refer to the table 11). This is supposed due to small content of silicon dioxide SiO_2. Each amount contained in serpentines is remarkably less that in cement, thus FP (fission product) in serpentines concrete is not likely to be more than that in normal aggregates concrete. Irradiation test by Suzuki et al. verified that serpentines concrete was stable up to 400°C radiation.

(2) Anti-Thermal Crushing Property of Serpentines

Serpentines before heating showed pretty different crushing resistance depending on their sources, which has relationship with abrasion loss of the aggregate and not with amount of crystalline water (refer to the table 3).

Crushing resistance of serpentines after heating is supposed to increase with temperature up to 300°C, and to continue to increase up to the temperature that crystalline water dissolves (refer to Fig. 2). The more crystalline water is contained, the more increase rate is. This phenomenon of increase is supposed to be due to consolidation of rock matrix resulting from heating or due to increase of internal friction resulting from decrease of water or due to decrease of pore pressure. Serpentines of large crushing resistance before heating showed also large value after heating.

When heated at 600°C, crushing resistance decreased by 35 ∿ 55% of initial values in some cases. Amount of crystalline water didn't influence this decrease of crushing resistance. This phenomenon is supposed to be due to brittle transformation of rock structure which is accompanied by the dissolution of crystalline water at 580°C heating.

Consequently heat resistance limitation of serpentines is supposed to be a temperature where crystalline water dissolves.

(3) Heat Resistance Properties of Serpentines Concrete

When serpentines concrete were brought in the atmosphere (20°C) after heating at 300 to 600°C, hair cracks occurred on the top surface (refer to photo 2). This is supposed because of thermal stress caused by rapid cooling of the surface, and cannot supposed to be peculiar phenomenon to serpentines concrete. Hair cracks developed only in case of heating at 600°C during leaving in the air thereafter. This can be explained as follows; Calcium compounds dissolves thermally into calcium oxide CaO at about 400°C and then calcium oxide changes into calcium hydroxide $Ca(OH)_2$ by absorbing moisture H_2O in the air and swells to cause hair cracks. This is also verified by that hair cracks occurred after heating at 300°C never developed. Consequently the practical limit of the temperature of the serpentines concrete employing normal portland cement may be determined to be below 400°C.

No difference of the compressive strength among concretes is observed before heating due to the sources of the aggregates (refer to the table 13). This is because all serpentines used have bigger strength than objective strength. After heating (refer to Fig. 3), aggregates used in case of 110°C drying and 300°C heating showed increase or decrease in strength which has inverse correlation with toughness of aggregates (guessed from the tables 3 and 12). There was however no effect of amount of crystalline water. The reasons of the strength fluctuation as shown in Fig. 8[9] are considered to be internal stress caused by the difference of the swelling rate in or shrinkage between cement paste and aggregates when heated and the increased strength of cement paste and aggregate resulting from heating and the canceling action of those two. As the aggregate which has small toughness is easy to follow the expansion and shrinkage of cement paste, less internal stress occurred and strength does not decrease. Strength of concrete when heated at 600°C decreases remarkably and its value is 150 ∿ 170 kg/cm^2 in average. There is no difference due to sources of aggregates. It may be probable that one of the reasons is deterioration in strength of aggregates themselves (refer to the table 11). However considering that the residual strength is almost even, it can be supposed to be caused by thermal deterioration of cement hydrate components. Consequently rather loose aggregates is advantageous to avoid deterioration in strength when heated at approximately 300°C. Difference depending upon amount of crystalline water is not evident.

Different tensile strength depending upon the source of the aggregate was given in each kind of concrete (refer to the table 14). The concrete containing such tough aggregate of which unit weight and crushing resistance are large and of which water absorption is high, shows high tensile strength. After heating (see to Fig. 4), tensile strength becomew lower by 0 ∿ 20% in case of 90°C drying and 300°C heating. The looser the aggregate is (e.g. Ube product), the smaller strength deterioration is. This is because the loose aggregate causes less internal stress due to heating as mentioned above. The aggregate which is pre-dried at 110°C and heated at 300°C shows larger deterioration in strength. This may be because of large shrinkage as mentioned below. The aggregate heated at 600°C remarkably deteriorates in strength, and the residual strength is 10 ∿ 11 kg/cm^2 (1/3 ∿ 1/4 of the initial value).

Effect of the locality of aggregates and predrying on tensile strength disappears. The phenomenon is supposed due to thermal deterioration of cement paste same as in case of compressive strength. Consequently it is practical to employ rather loose aggregate to increase the residual tensile strength when heated at around 300°C. Amount of crystalline water does not seem to effect on the tensile strength.

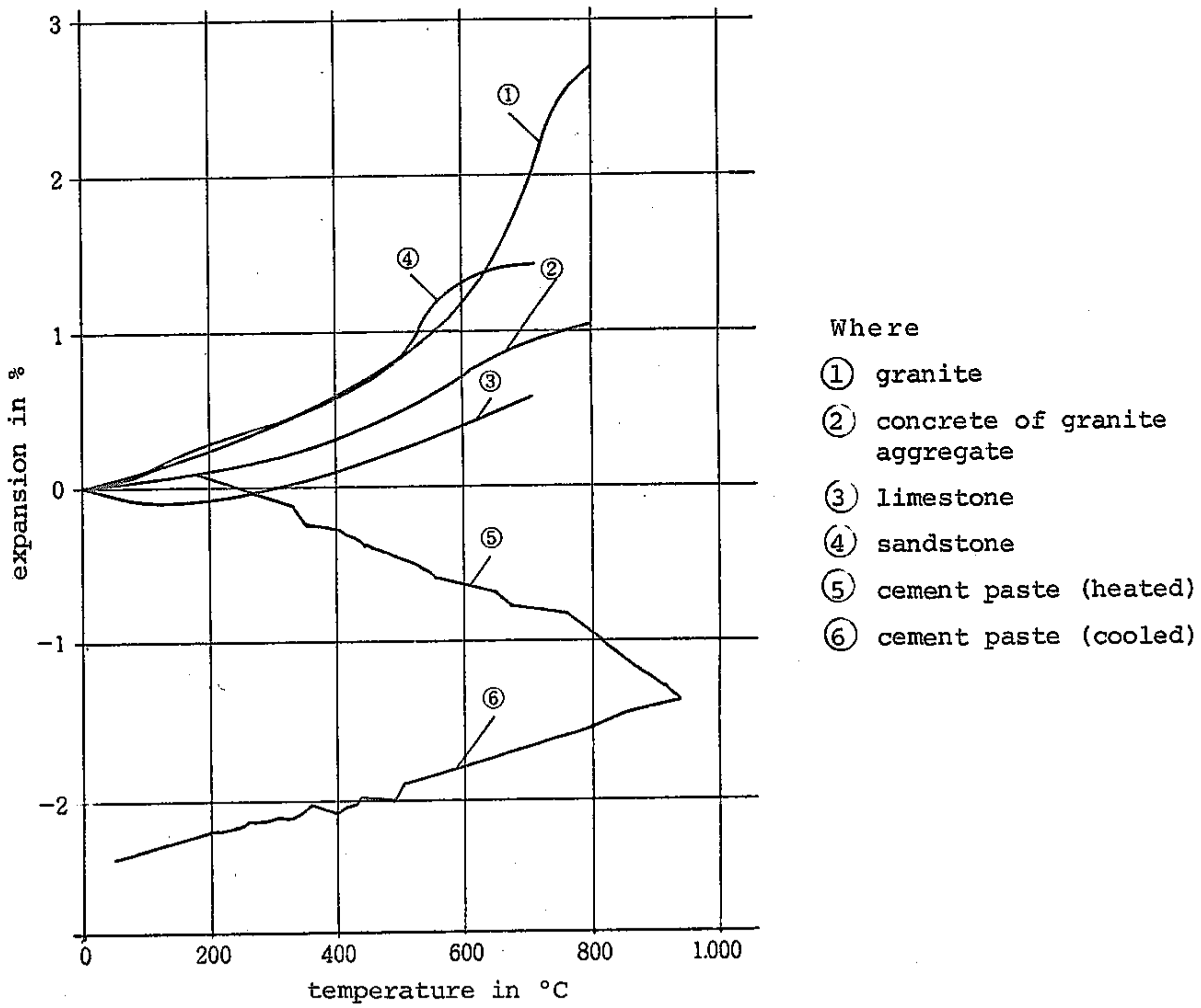

Fig. 8 Relationship between temperature and swelling of aggregates and cement

Difference of the dynamic modulus of elasticity of concrete before heating depending upon the sources of aggregates was recognized. The loose aggregate (Ube product) shows small value (refer to the table 15). When predried at 110°C, the modulus decreases by 12 ∿ 17%, but does not differ from one aggregate to another. When heated at 300°C, the modulus decreases by 49 ∿ 61%, and differs slightly from one aggregate to another; decreasing rate of the concrete using aggregate which contains less crystalline water in quantity (Miyamori product) is slightly more than others. Dynamic modulus of elasticity may be effected by the water content. When heated at 600°C, it decreases by 91 ∿ 94%, and the residual modulus is 17 ∿ 28 x 10^3 kg/cm^2. Difference depending upon the sources of aggregates dissappears. This phenomenon seems to be the result of the following; crystalline water in the aggregates and calcium compounds in cement has almost desiccated, thus most water in the material got scattered and lost. Consequently the more crystalline water is contained in the aggregate, when heated at about 300°C, the more decrease of dynamic modulus of elasticity is restricted. No obvious difference in elongation of concrete is shown from one aggregate to another during curing before heating (refer to the table 16). After drying and heating (refer to Fig. 6), concrete shrinks by 0.07 ∿ 0.17% after predrying at 110°C and by 0.20 ∿ 0.45% after heating at 300°C depending upon the sources of aggregates. The looser aggregate (Ube product) shows large elongation. The reason is as follows, as mentioned above; the looser aggregate causes less internal stress. Concrete heated at 300°C after predrying shows slightly larger shrinkage, which seems to result from advanced desiccation as mentioned below. Concrete heated at 600°C shrinks 3 to 4 times compared with that heated at 300°C, and shows remarkable difference from one source of the aggregate to another. As reasons of the phenomenon, the shrinkage due to desiccation of calcium compounds in cement at around 400°C, the dissolution of crystalline water in the aggregate and the passive shrinkage of loosened aggregates affected by the deformation of the cement paste are to be considered. Toughness of the aggregate is supposed to give difference in quality of the concrete in this case. Consequently tough aggregate is desirable to get concrete of less shrinkage.

Weight of concrete decreases by 0.2 ∿ 1.8% during curing before heating (refer to the table 17). An abnormal phenomenon was observed that the aggregate containing larger quantity of crystalline water showed larger amount of desiccation inversely proportional to amount of absorbed water by the aggregate. Weight of concrete reduces by 5.0 ∿ 6.5% after drying at 110°C, and difference depending upon the sources of aggregates was observed. The more the absorbed water is, the more weigh loss is observed. Concrete heated at 300°C reduces in weight by 6.5 ∿ 8.5% which is larger than dried at 110°C and which depends on the locality of the aggregates. The reasons of weight loss are supposed complete desiccation of free water in the aggregate and partial desiccation of hydrated water of the cement. The amount reduced is more than that of absorbed water in the aggregate, so that not only amount of free water content but also the air void of the aggregate gives influence for evaporation the water. Weight reduces by 15.3 ∿ 18.6%, when heated at 600°C remarkably larger than heated at 300°C, and the difference due to the sources of the aggregates is observed. The weight loss in this case is caused also by evaporation of hydrated water of calcium compounds in cement (at about 400°C) and of crystalline water of the aggregate (at about 580°C). The amount shows close relation with amount of the crystalline water and the air void of the aggregate.

Consequently the aggregate which is low absorption of water and has large quantity of crystalline water is desirable to retain sufficient water in concrete when in the duty at 300°C in temperature.

Effect of heating period appears in a different way for the kind of properties. The properties relating strength which are remarkably sensitive for internal stress and thermal dissolution deteriorate or become stable in the beginning of heating. On the contrary, properties relating volume and weight which are influenced remarkably by desiccation of water suffer effect of continuous heating. The more absorptive and the looser the aggregate is, the less effect of continuous heating is.

6. CONCLUSION

Results are summarized as follows considering all tests mentioned above.

(1) As minute components, Uranium and Thorium were detected from serpentines. The former was 0.1 to 0.2 ppm in weight, and the latter 0 to 1 ppm. The quantity was found to be remarkably less than that contained in cement. Consequently serpentines are supposed not to be harmful if employed as aggregates of concrete.

(2) Heat resistant crushing value of serpentines were found to increase by 20 to 50% by heating at 300°C and all serpentines were fully heat resistant around up to the temperature of dissolution of crystalline water. However the value when heated at 600°C was found to decrease by 30 $\sim$ 50%.

(3) Tensile and compression strength of serpentines concrete increased or decreased by drying and heating at 90 $\sim$ 300°C. The looser aggregates showed higher residual strength than tough aggregates. It was found that there was no relation between quantity of crystalline water contained and deterioration of strength. Consequently the aggregate which is loose to some extent and contains more crystalline water is desirable in point of strength and shield performance. However, when heated at 600°C, all concrete was found to remarkably deteriorate in strength and qualitative difference come from the original source of the aggregates disappeared.

(4) Dynamic modulus of elasticity of serpentines concrete was revealed to decrease by 45 to 60% counter-proportionally to amount of crystalline water in the aggregate when heated

at 300°C. Consequently the aggregate abundant in crystalline water is desirable to get higher dynamic modulus of elasticity. However it was also found that every material lost dynamic modulus of elasticity when heated at 600°C.

(5) Shrinkage of serpentines concrete due to drying and heating was 0.2 ∿ 0.45% when heated at 300°C. The loose aggregate showed larger shrinkage. Consequently the tough aggregate is desirable to prevent shrinkage. When heated at 600°C, this increased three to four times of above value and it was found that concrete is more shrinkable in case of the looser aggregate.

(6) Weight loss of serpentines concrete heated at 300°C was 6.5 to 8.6%. It was found that those composed of the aggregate of larger water absorption showed larger weight loss, and that the deviation of the loss was superior than that of absorbed water. Consequently the aggregate containing less absorbed water is desirable to prevent weight loss. When heated at 600°C, remarkable loss was found, and the aggregate containing larger amount of absorbed and crystalline water showed the larger weight loss.

(7) As long as relationship between heating period and stabilization period of concrete's properties is concerned, the tough aggregate takes more time to be stable than the loose aggregate. Time required for stabilization was also found to depend on kinds of properties.

(8) Concrete was stable during heating, however when exposed in the air just after heating and cooled rapidly, hair cracks were appeared on the top surface of concrete. When heated at 300°C, they didn't develop further. However it was found to develop gradually when heated at 600°C.

As mentioned above all kinds of serpentines here treated contain negligible amount of FP components and are heat resistant by themselves up to considerably higher temperature, which were revealed by crushing test. Generally speaking properties of serpentines concrete deteriorate by heating except some properties of the aggregates, and their extent depends mainly on toughness or looseness of the aggregates. Consequently it is recommended to select the aggregates those containing large quantity of crystalline water, being the purpose of application taken into consideration.

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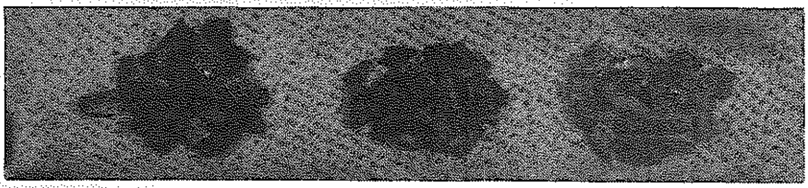
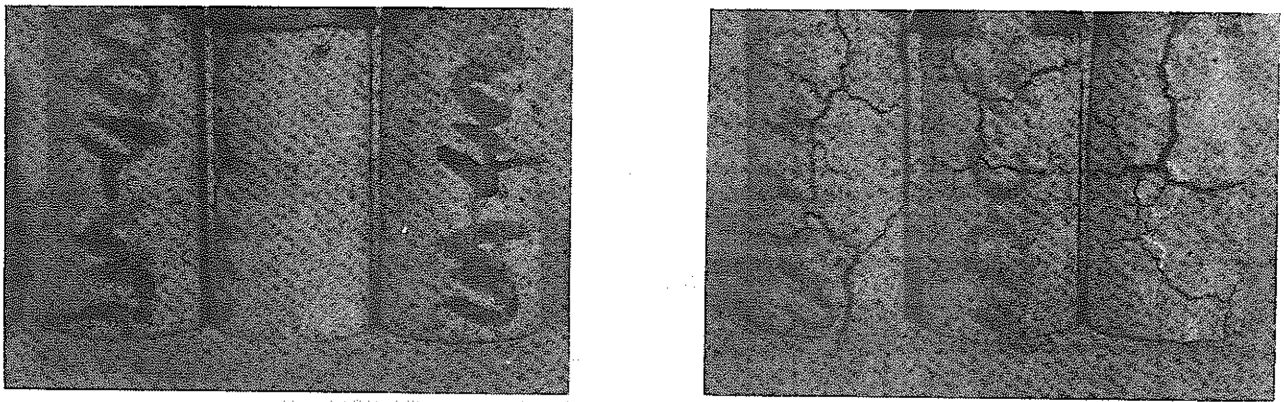


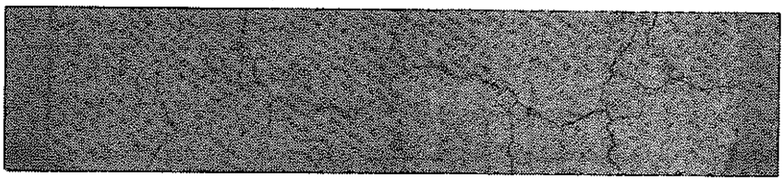
Before heating at 20°C | After heating at 300°C | After heating at 600°C

Photo 1 Color of serpentines before and after heating

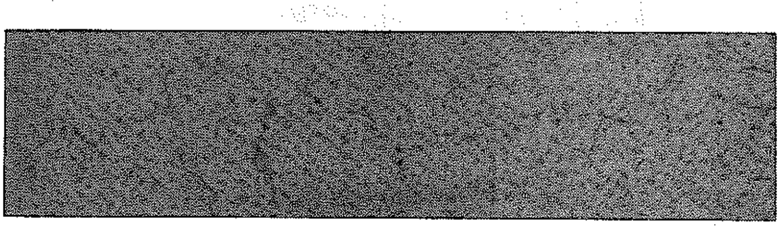


Heated at 300°C, left in the air for 20 days (Ube) | Heated at 600°C, left in the air for 20 days (Ube)

Photo 2a Appearance of concrete after heating (ø15 x 30cm)



Heated at 600°C, left in the air for 20 days (Ube)



Heated at 300°C, left in the air for 20 days (Ube)

Photo 2b Appearance of concrete after heating (10x10x40 cm)

Appendix table 1 An estimated and produced amounts of serpentines used for test

Name of serpentines	Miyamori Product	Ube Product	Ogose Product
Locality	Miyamori-village, Iwate Prefectur	Ube-city, Yamaguchi Prefecture	Ogose-cho, Saitama prefecture
stimated amount in ton	1,500,000	3,200,000	1,500,000
Average amount of production	13,000	60,000	10,000
Maximum amount of production	40,000	100,000	15,000
Main applications	① Chemical manure ② Raw material for cement ③ Quarrying (for gardering)	① Aggregate for concrete ② Sub raw material for iron manufacture ③ Chemical manure ④ Raw material for light aggregate	① Aggregate for road ② Small quantity, for others
Numbers of employee	-	4	10
Quarrying manufacturer	K.K. Toyo Soda	K.K. Edamura Corp.	K.K. Tamura Stone