Internal standard material: rutile type titanium dioxide

[0048]

Rietveld Analysis Conditions

Rietveld analysis software: TOPAS Ver. 4.2 (manufactured by Bruker AXS GmbH)

Zero-point correction: not performed

Correction of height of sample surface: performed

Analysis target mineral: quartz, mullite (3:2), anhydrite, limestone, magnetite, hematite, and titanium dioxide (only a sample added as an internal standard material) Preferred orientation function of hematite phase: assuming that preferred orientation of the hematite phase appears on a diffraction line of the (110) plane at a diffraction angle 2θ of about 35.5°, refinement was performed using a March-Dollase function with an initial coefficient value as 1. Regarding the magnetite phase, it was assumed that preferred orientation did not appear.

[0049]

The measurement procedure of the crystal phase such as magnetite or hematite and the amorphous phase in the fly ash was as described below.

(i) As the internal standard material, fly ash (sample 1) to which 20 mass% of rutile type titanium dioxide was added, and fly ash (sample 2) to which no internal standard material was added were prepared.

(ii) The fly ash (sample 2) to which no internal standard

following Expression (6).

Unburned Carbon having Particle Diameter of more than 212 μm/Unburned Carbon (%)=Unburned Carbon (mass%) having Particle Diameter of more than 212 μm in Fly Ash÷Unburned Carbon (mass%) in Fly Ash×100 (6)

[0054]

Cement Composition

(Examples 1 to 7, Reference Examples 8 to 10)

The fly ashes prepared in Preparing Examples 1 to 7 were mixed with normal Portland cement at mixing ratios shown in Table 2, respectively. As a result, cement compositions according to Examples 1 to 7, and Reference Examples 8 to 10 were manufactured.

[0055]

(Comparative Examples 1 to 6)

The fly ashes prepared in Reference Preparing Example 1 and Comparative Preparing Examples 2 to 6 were mixed with normal Portland cement at mixing ratios shown in Table 2, respectively. As a result, cement compositions according to Comparative Examples 1 to 6 were manufactured.

[0056]

Regarding each of the obtained cement compositions, the mass ratio (%) of the unburned carbon having a particle diameter of more than 212 μm in the cement composition, fluidity, mortar compressive strength, and setting time were measured.

[0061]

[Table 2]

		Fly Ash								Cement Composition								
														Setting Time Test		Mortar Compressive Strength		
		Use Ratio of Fly Ash to Raw Material (%)	Blaine specific surface area (cm^2/g)	Particle Diameter Ratio (D50-D10)/(D90-D50)	Amount of Amorphous Phase (mass%)	Amount of Fe in Amorphous Phase (mass%)	Amount of Unburned Carbon (mass%)	Amount of Unburned Carbon having Particle Diameter of more than 212 μm (mass%)	Mass Ratio Amorphous Phase/ Unburned Residue (%)	Fly Ash Content (mass%)	Amount of Unburned Carbon having Particle Diameter of more than 212 μm (mass%)	Flow Value (20°C) (mm)	Flow Value (30°C) (mm)	Initial Setting Time (min)	Final Setting Time (min)	3 Days (N/mm^2)	28 Days (N/mm^2)	91 Days (N/mm^2)
Example 1	Preparing Example 1	100	3210	0.32	75.1	3.6	5.0	0.5	10.0	15	0.1	193	174	110	165	29.5	56.2	79.5
Example 2		100	3210	0.32	75.1	3.6	5.0	0.5	10.0	25	0.1	194	174	120	185	24.1	51.7	79.2
Example 3	Preparing Example 2	100	3500	0.38	59.8	6.0	9.5	2.4	25.3	20	0.5	189	171	130	190	26.8	54.3	77.5
Example 4		100	3500	0.38	59.8	6.0	9.5	2.4	25.3	30	0.7	190	171	135	195	22.6	51.2	79.8
Example 5	Preparing Example 3	100	4300	0.47	65.3	4.4	14.8	4.5	30.4	20	0.9	185	167	140	200	26.5	54.3	76.3
Example 6		100	4300	0.47	65.3	4.4	14.8	4.5	30.4	29	1.3	184	166	155	210	23.0	50.5	78.1
Example 7	Preparing Example 4	100	3210	0.47	61.3	6.5	5.0	0.9	18.0	11	0.1	180	166	75	135	29.5	59.2	78.1
Reference Example 8	Preparing Example 5	100	4100	0.35	65.8	3.3	7.0	0.9	12.9	25	0.2	182	169	135	170	22.5	47.9	72.2
Reference Example 9	Preparing Example 6	100	2800	0.27	71.5	3.7	5.5	0.9	16.4	25	0.2	196	168	120	170	23.8	47.5	73.8
Reference Example 10	Preparing Example 7	100	3520	0.25	51.5	9.5	14.8	2.3	15.5	25	0.6	185	173	155	195	22.3	48.8	71.5
Comparative Example 1	Reference Preparing Example 1	70	3900	0.17	61.5	4.8	1.9	0.1	5.3	25	0.0	175	162	115	165	24.4	53.8	75.5
Comparative Example2	Comparative Preparing Example 2	100	3150	0.18	65.1	5.8	14.5	5.2	35.9	28	1.5	175	145	160	215	23.2	47.1	73.2
Comparative Example3	Comparative Preparing Example 3	100	3210	0.47	67.3	5.7	5.0	0.9	18.0	33	0.3	185	170	145	220	20.5	47.1	75.2
Comparative Example4	Comparative Preparing Example 4	100	4400	0.19	55.1	6.5	15.5	3.0	19.4	28	0.8	176	148	155	205	23.0	47.3	73.2
Comparative Example5	Comparative Preparing Example 5	100	3800	0.14	70.1	4.4	14.9	5.2	34.9	30	1.6	168	145	160	215	22.6	47.3	75.1
Comparative Example6	Comparative Preparing Example 6	100	4650	0.51	71.5	6.4	13.0	2.3	17.7	25	0.6	175	140	100	155	22.8	50.2	79.1

[0062]

As shown in Table 2, in the cement compositions of Examples 1 to 8 using the fly ashes of Preparing Examples 1 to 5 in which the ratio (D50-D10)/(D90-D50) was higher than 0.3 and 0.5 or lower, the flow value at 20°C was superior at 180 mm or higher, the flow value at 30°C was superior at 165 mm or higher, and both the initial mortar compressive strength at a material age of 3 days and the mortar compressive strength at a material age of 28 days were high. In addition, in the cement compositions of Examples 1 to 7, the long-term mortar compressive strength at a material age of 91 days was high at 76.3 N/mm^2 or higher, and long-term strength development was further improved.

[0063]

In the cement composition of Example 7, the fly ash content of Preparing Example 4 in the cement composition was low at 11 mass%. Therefore, the fluidity and the compressive strength were superior, but the setting time was shorten.

[0064]

In the cement composition of Reference Example 8, the fly ash of Preparing Example 5 was used, and the amount of Fe in the amorphous phase of the fly ash of Preparing Example 5 was low at 3.3 mass%. Therefore, the long-term strength development at a material age of 28 days and a material age of 91 days was lower.

[0065]

In the cement composition of Reference Example 9, the fly ash of

Preparing Example 6 was used, the Blaine specific surface area of the fly ash of Preparing Example 6 was 2800 cm^2/g, the amount of coarse powder was relatively large, and the long-term strength development at a material age of 28 days and a material age of 91 days was lower.

[0066]

In the cement composition of Reference Example 10, the fly ash of Preparing Example 7 was used, and the amount of the amorphous phase in the amorphous phase of the fly ash of Preparing Example 7 was low at 51.5 mass%. Therefore, the long-term strength development at a material age of 28 days and a material age of 91 days was lower.

[0067]

As shown in Table 2, in the fly ash of Reference Preparing Example 1 which was the same as Type II fly ash defined in JIS A6201 "Fly Ash for Use in Concrete", about 30 vol% of the fly ash was necessarily removed from the raw material fly ash in order to satisfy the JIS standard, the ratio of the fly ash used to the raw material fly was 70%, and the entire amount of the raw material fly ash was not efficiently used.

[0068]

As shown in Table 2, in the cement composition of Comparative Example 1, the ratio (D50-D10)/(D90-D50) of the fly ash of Reference Preparing Example 1 was low at 0.17, and the initial mortar compressive strength at a material age of 3 days,

CLAIMS

1. A cement composition which comprises cement and fly ash, and a fly ash content is 30 mass% or lower, and the fly ash satisfies the following Expression (I) in a volume-based particle diameter distribution, the amount of an amorphous phase in the fly ash is 55 mass% or higher and 75.1 mass% or lower with respect to the total amount of a crystal phase and the amorphous phase in the fly ash, the amount of Fe in the amorphous phase of the fly ash is 3.5 mass% or higher and 10 mass% or lower, and a Blaine specific surface area of the fly ash is 3000 cm^2/g or higher and 4500 cm^2/g or lower,

$$0.25 \leq (D50-D10)/(D90-D50) \leq 0.47 \quad (I)$$

(where D10, D50, and D90 represent particle diameters corresponding to a cumulative frequency of 10%, a cumulative frequency of 50%, and a cumulative frequency of 90% in order from the smallest diameter of the fly ash).

2. The cement composition according to claim 1, wherein the fly ash content is 12 mass% or higher.

3. The cement composition according to any one of claims 1 or 2, wherein the amount of unburned carbon in the fly ash is 3 mass% or higher and 15 mass% or lower.

4. The cement composition according to any one of claims 1 to 3, wherein the amount of unburned carbon having a particle diameter of more than 212 μm in the cement composition is 1.5 mass% or lower.

5. The cement composition according to any one of claims 1 to 4, wherein a mass ratio of the unburned carbon having a particle diameter of more than 212 μm in the fly ash to the unburned carbon in the fly ash is 35 mass% or lower.

6. A method for preparing fly ash for a cement composition, the method comprising: classifying raw material fly ash, crushing the classified coarse powder fly ash having a particle diameter of 45 μm or more, and mixing the raw material fly ash and the crushed fly ash with each other so as to satisfy the following Expression (I) in a volume-based particle diameter distribution, the amount of an amorphous phase in the mixed fly ash is 55 mass% or higher and 75.1 mass% or lower with respect to the total amount of a crystal phase and the amorphous phase in the mixed fly ash,

the amount of Fe in the amorphous phase of the mixed fly ash is 3.5 mass% or higher and 10 mass% or lower, and a Blaine specific surface area of the fly ash is 3000 cm^2/g or higher and 4500 cm^2/g or lower,

$$0.25 \leq (D50-D10)/(D90-D50) \leq 0.47 \quad (I)$$

(where D10, D50, and D90 represent particle diameters corresponding to a cumulative frequency of 10%, a cumulative frequency of 50%, and a cumulative frequency of 90% in order from the smallest diameter of the fly ash respectively).

7. A method for producing a cement composition, the method comprising: classifying raw material fly ash, crushing the classified coarse powder fly ash having a particle diameter of 45 μm or more, mixing the raw material fly ash and the crushed fly ash with each other so as to satisfy the following Expression (I) in a volume-based particle diameter distribution, and adding the mixed fly ash such that the amount thereof is 30 mass% or lower with respect to the total amount of the cement composition, the amount of an amorphous phase in the mixed fly ash is 55 mass% or higher and 75.1 mass% or lower with respect to the total amount of a crystal phase and the amorphous phase in the mixed fly ash, the amount of Fe in the amorphous phase of the mixed fly ash is 3.5 mass% or higher and 10 mass% or lower, and a Blaine specific surface area of the fly ash is 3000 cm^2/g or higher and 4500 cm^2/g or lower,

$$0.25 \leq (D50-D10)/(D90-D50) \leq 0.47 \quad (I)$$

(where D10, D50, and D90 represent particle diameters corresponding to a cumulative frequency of 10%, a cumulative frequency of 50%, and a cumulative frequency of 90% in order from the smallest diameter of the fly ash respectively).

8. The method for producing a cement composition according to claim 7, wherein the mixed fly ash is added such that the amount thereof is 12 mass% or higher with respect to the total amount of the cement composition.