Pergamon

Cement and Concrete Research 29 (1999) 241–247

CEMENT AND CONCRETE RESEARCH

Microwave curing effects on the 28-day strength of cementitious materials

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Manuscript received 4 February 1998; accepted manuscript 14 July 1998

Abstract

Microwave energy can be applied on the curing of the cementitious materials because of their water content. Isothermal heating, achieved best by feedback temperature control, is required to investigate properly the temperature effects on the properties. Type I Portland cement mortars, some with additions of appropriate amounts of pozzolanic material, such as slag, silica fume, or class F fly ash, were cured isothermally with feedback temperature control at several temperatures. The curing times were determined by the instrumented penetration test, and 28-day compressive strengths were measured. Optimum curing conditions were found for 40°C and 60°C, but 80°C was unsuitable for microwave curing.

Keywords: Microwave processing; Mechanical properties; Mortar; Temperature

It is important in the precast concrete process to reduce the curing period for the productivity, capital saving, and reduction of workshop area. To satisfy this demand, the use of an external heating source has been investigated. The steam curing process, which operates at atmosphere pressure and lower than 100°C, accelerates cement hydration and makes the curing time shorter [1]. Thus, steam curing can offer the precast industry an attractive substitute for normal curing. Unfortunately, steam curing has some drawbacks; although steam cured specimens show a great increase in early strength, long-term strength generally is lower than specimens cured under normal conditions [2–5]. The steam curing process relies on the thermal conductivity of the specimen, with heat flowing from the exterior to the interior. The larger the material, the slower the heat penetration to the interior and the less uniform the heating throughout the material.

High-frequency electromagnetic heating, such as microwave enhanced heating, is able to reduce such nonuniformity due to its superior penetration depth [6]. Because microwave energy generates heat in cementitious materials due to the dielectric nature of the water, microwave enhanced heating is a potential alternative method for accelerating cement hydration. Microwave processing has several advantages over steam curing in the precast concrete industry. First, microwave energy can heat a specimen uniformly and volumetrically, being independent of the thermal conductivity of the specimen. Second, it can more easily enhance the evaporation rate and better control energy absorption, optimizing the overall heating process before demolding. Last, the final performance of the cement-based materials can potentially be improved to achieve long-term goals [7].

Previous reports of the effect of microwave curing on long-term strength are inconsistent. This may be related to uncertainties in the temperatures. The pioneering work of Watson [8] shows that 28-day compressive strength of microwave cured specimens displayed only half the strength of the normally cured specimens. However, his results were uncertain because the temperature of specimens might have fluctuated due to the pulsed microwave energy he was using. Also, an internal temperature of 90°C was reached, at which temperature cracks could be generated as a result of escape of steam from the interior [7]. Wu and coworkers [9] reported that microwave curing improved the 28-day compressive strength of mortar as high as 3% to 7% as well as enhancement of short-term strength. They emphasized the process optimization of the internal temperature and the final water-to-cement ratio of the specimen, controlling processing time, and microwave power. Too much microwave energy could cause a decrease of strength due to overevaporation and overheating.

Other independent results confirmed the possibility of improving the 28-day compressive strength by microwave

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PII: S0008-8846(98)00189-6

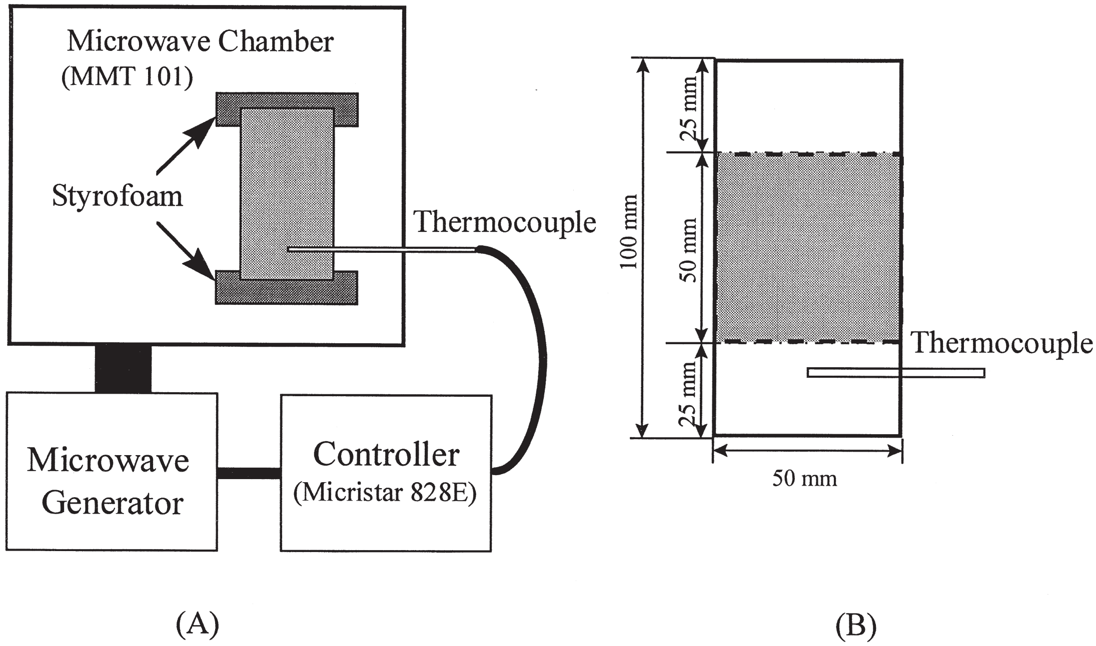

Fig. 1. (*A*) Schematic diagram of the microwave oven (not to scale). (*B*) Dimensions of the mortar specimens. The gray region was used for strength tests.

processing [7,10,11]. Recently, Leung and Pheeraphan [12,13] reported that the microwave curing technique can potentially produce concrete with very high early strength and little deterioration in its long-term performance by controlling material compositions and microwave power. In a subsequent study, they found the most important parameter to control was specimen temperature during the curing process. They accomplished temperature control by either a feedback temperature control or discrete power application based on the data from the feedback temperature control. Both methods can reduce overheating and thermal shock during curing [14]. The effect of the microwave enhanced heating process on cementitious materials needs more careful and precise work that includes the monitoring of process variables that will affect the properties of the final products.

One shortcoming of previous studies is uncertainty about the thermal history of the specimen during curing; typically, the power, rather than the temperature, was fixed. Isother-

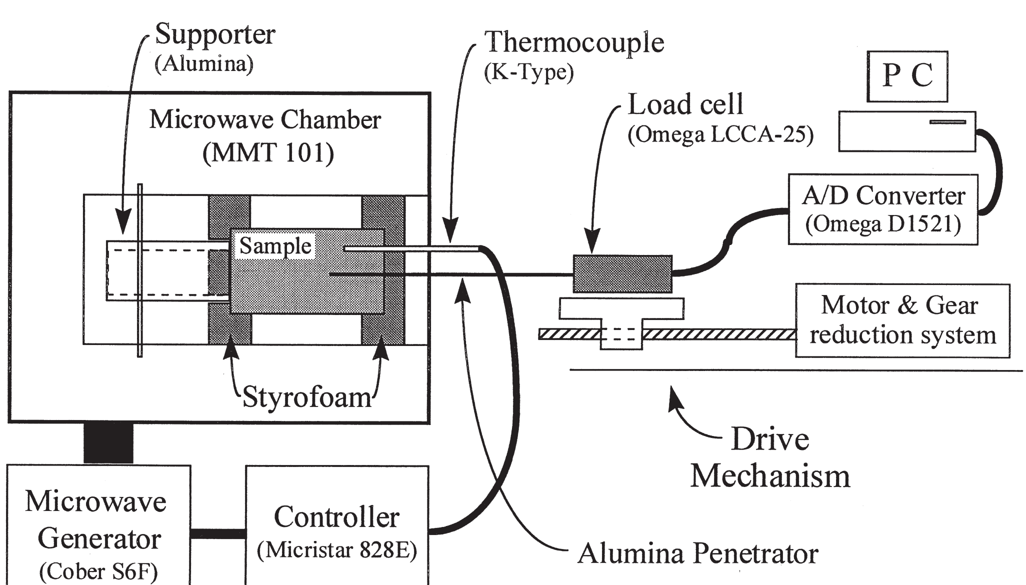

Fig. 2. Schematic diagram of the instrumented penetration test unit and the microwave oven (not to scale).

mal heating, achieved best by feedback temperature control, is required to properly investigate the temperature effects on the properties.

1. Experimental procedure

A highly overmoded cylindrical microwave oven (MMT 101, Oak Ridge, TN; 750 mm in diameter and 1200 mm long) with a power output range of 0 to 6 kW was used for the curing process of cement pastes and mortars. Temperature was measured by a shielded type-K thermocouple inserted directly into the specimen. The thermocouple output was fed to a controller that regulated the power input to the oven (Fig. 1). The heating rate was fixed at 7°C/min. The control thermocouple was placed 22 mm from the bottom of the specimen, out of the portion to be used in strength testing. Additional thermocouples were placed in some specimens at 37 and 67 mm from the specimen bottom to determine temperature uniformity during heating.

For mortar specimens, ordinary Portland cement (OPC) type I (Holnam, Clarksville, MO) and ASTM C778 graded silica sand [15] (AGSCO, Wheeling, IL) were used for this study. To investigate 28-day strength of blended mortars, 50% in weight ground granulated blast furnace slag, 10% silica fume, or 20% class F fly ash was substituted. The cementitious-to-sand ratio and deionized water-to-cementitious ratio for all mortar specimens were fixed at 1:2 and 0.4, respectively. The specimens were mixed for 10 min using a planetary-type mixer and then cast into a cylindrical polyethylene container 50 mm in diameter and 100 mm long. The specimen was vibrated to eliminate air bubbles and then capped with polyethylene covers. The application of microwave energy began 30 min after the water was added to the mix and then varied in accordance with the setpoint temperature, which was raised at a constant heating rate of 7°C/min to each preset curing temperature and maintained at that temperature for 2 h.

After curing, the specimens were stored in 100% relative humidity containers for 1 day, then removed from the mold and immersed in saturated lime water at 20°C for 27 more days until testing at 28 days. As a reference, normal cured specimens also were prepared. Both normal cured and microwave cured specimen were cut 25 mm from both ends and ground before testing. The final dimension of the strength test specimen was 50 mm $\times$ 50 mm (Fig. 1B; gray region).

To determine the curing time, an instrumented penetration test was employed (Fig. 2). As an alumina flat-tip penetrator of 2.8-mm diameter was driven into the specimen at a constant velocity by the drive mechanism, penetration hardness development was measured by the load cell (Omega LCCA-25, Stamford, CT; 25 lbf max) at the end of the penetrator. Details of the instrumented penetration test are given elsewhere [16].

2. Results and discussion

The three thermocouples in preliminary specimens demonstrated good temperature uniformity. Fig. 3 shows the temperature profiles measured by these thermocouples at the set temperatures of 40°C, 60°C, and 80°C. At the beginning, the temperature differences were about 1°C to 4°C among the three thermocouples, being larger at the higher temperature. However, these differences were considered to be too small to be significant. Overheating was not found. Thus, all of the specimens could be cured isothermally at the proposed curing temperature used in this study.

Two hours was arbitrarily chosen for the microwave heating time. Fig. 4 shows the 28-day compressive strength of type I and 50% slag mortars under 2-h heating by micro-

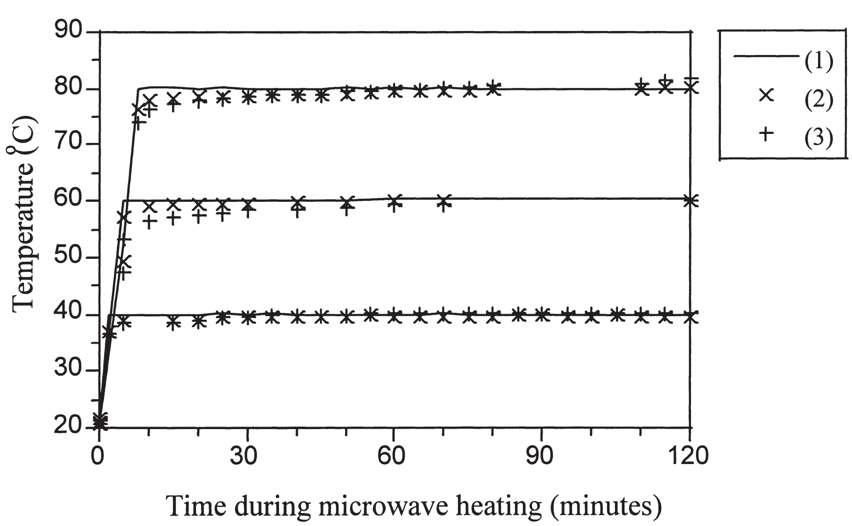

Fig. 3. Temperature histories for the mortar specimens measured by thermocouples placed (1) 22 mm, (2) 37 mm, and (3) 67 mm from the bottom of the specimen at curing temperatures of 40°C, 60°C, and 80°C.

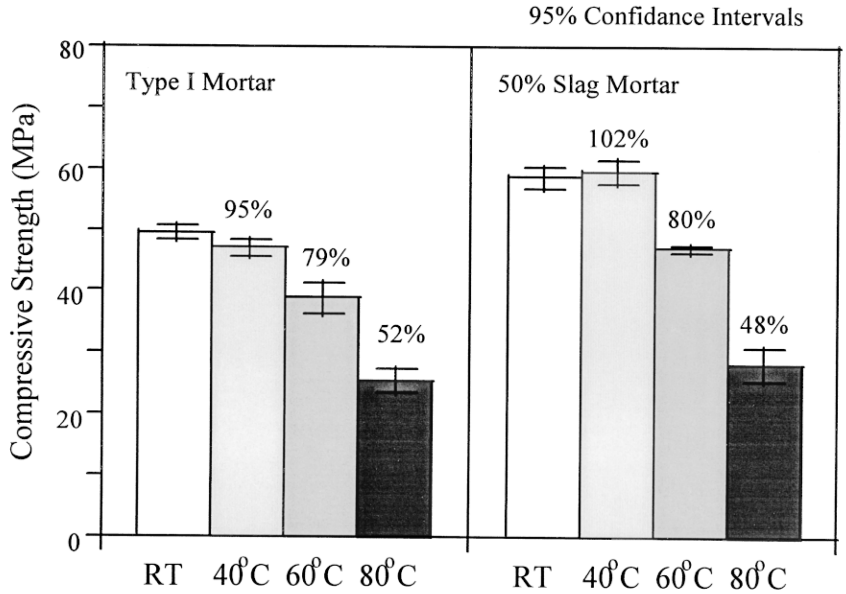

Fig. 4. The 28-day compressive strength for room temperature cured and microwave cured OPC type I and 50% slag mortars at 40°C, 60°C, and 80°C with sand-to-cement ratio = 2:1, water-to-cement ratio = 0.4, and heating time = 2 h. RT: room temperature.

wave energy at various temperatures. The 95% confidence intervals based on three identical specimens are superimposed on the average value. At 40°C, the compressive strengths of both cement mortar and 50% slag mortar are similar to those cured at room temperature, considering the confidence intervals. In contrast, specimens cured at 80°C have only half of the room temperature strengths for both. In the case of 60°C, specimens show 80% strength of the room temperature cured specimens.

However, it was thought that 2 h may be unnecessarily long for heating, and that less strength degradation might be realized for shorter heating times. To explore this, the instrumented penetration test was introduced. This test monitors hardness development during hydration, with typical results shown in Fig. 5. Three levels of the penetration hardness, namely, 1, 5, and 13 MPa, were chosen arbitrarily to establish the heating time for all curing temperatures. Table 1 indicates the time needed to reach each level of penetration hardness for cement mortar at the various curing temperatures. Specimens for strength tests were cured according to Table 1. In other words, as an example, at 1 MPa and 40°C, the specimen was heated 76 min at 40°C, and then allowed to cool naturally to room temperature.

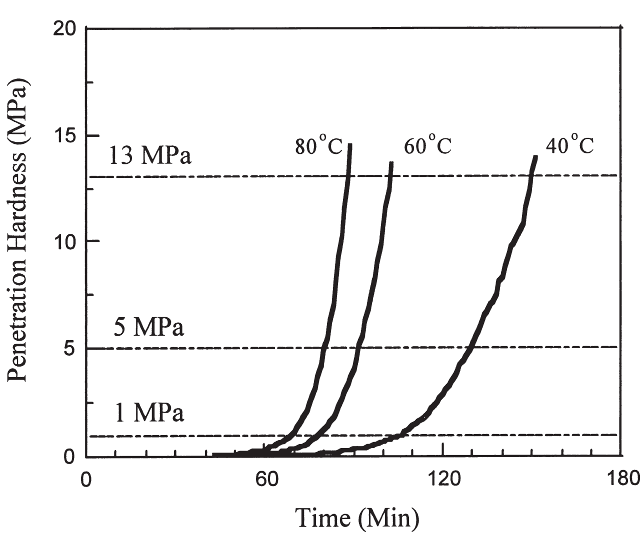

Fig. 5. Penetration hardness development curves at 40°C, 60°C, and 80°C obtained by the instrumented penetration test.

Fig. 6 shows the 28-day strength of cement mortars heated at 40°C, 60°C, and 80°C until the penetration hardness reached each stated value, as shown in Table 1. The results of curing at 40°C or 80°C are very close to the 2-h curing results shown in Fig. 4. The strength values are unchanged under 40°C curing temperature until 13 MPa was reached, but at 80°C they are drastically decreased. However, the behavior at 60°C curing is different. When specimens were cured at 60°C until the penetration hardness reached 1 MPa, or 48 min of curing time for cement mortar,

Table 1
Microwave heating time of cement mortar at various curing temperatures to reach given levels of penetration hardness

Penetration hardness	Curing temperature		
	40°C	60°C	80°C
1MPa	76 min	48 min	39 min
5MPa	100 min	62 min	50 min
13MPa	120 min	73 min	58 min

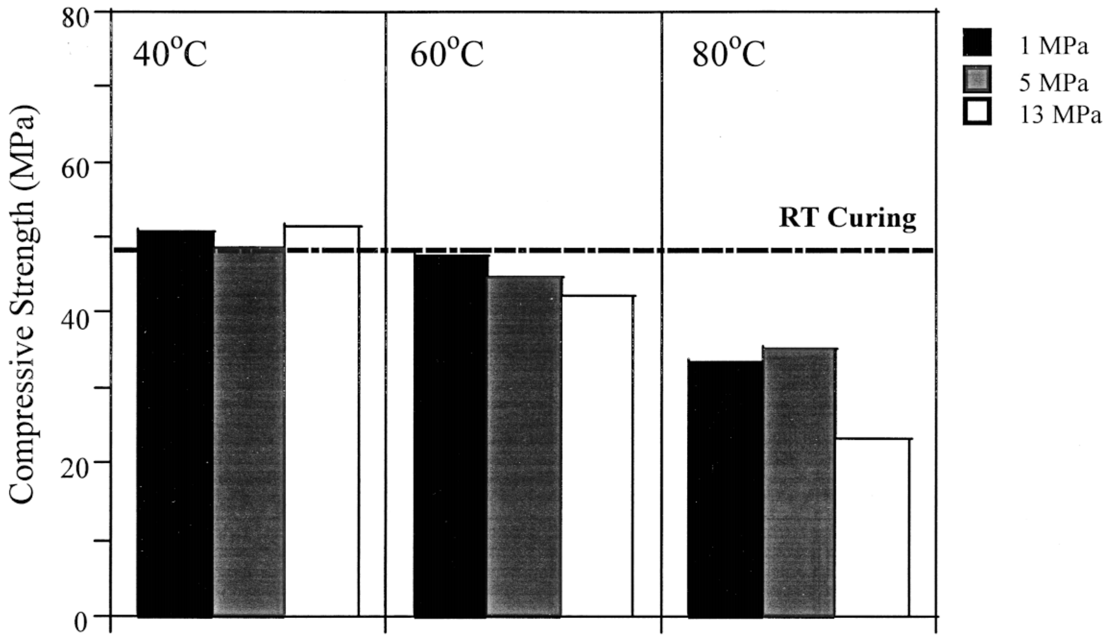

Fig. 6. The 28-day compressive strength for room temperature cured and microwave cured OPC type I mortars at 40°C, 60°C, and 80°C with sand-to-cement ratio = 2:1 and water-to-cement ratio = 0.4. Specimens were heated until the penetration hardness reach 1, 5, or 13 MPa.

their 28-day strength was unchanged compared with that of room temperature cured specimens, which is indicated by the dotted line on the same figure. As the heating time was longer than 48 min (or 1 MPa), the strength decreased. Although the strength decreased to 88% of room temperature at 13 MPa (73 min), it still is higher than that of the 2-h cured specimens (79%) shown in Fig. 4. From this study, the best curing times for cement mortar without degradation of 28-day compressive strength are 120 min (or 13 MPa) at 40°C and 48 min (or 1 MPa) at 60°C.

To estimate the time reduction to demold cement mortar, the final setting time, defined by ASTM C403 [17], when

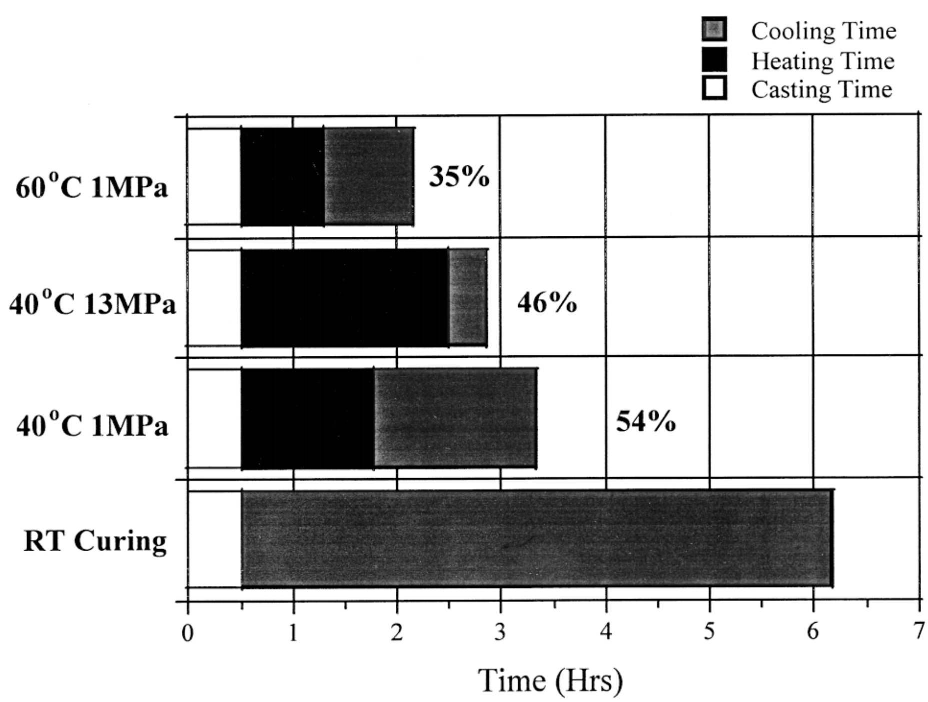

Fig. 7. Final setting times for room temperature cured and microwave cured OPC type I mortars with sand-to-cement ratio = 2:1 and water-to-cement ratio = 0.4.

Table 2
Microwave heating time of blended mortars at several curing temperatures to reach each level of penetration hardness

	Penetration hardness	Curing temperature		
		40°C	60°C	80°C
50% slag	1MPa	105 min	53 min	39 min
	5 MPa	141 min	72 min	52 min
	13 MPa	172 min	88 min	61 min
10% silica fume	1 MPa	60 min	39 min	33 min
	5 MPa	90 min	56 min	46 min
	13 MPa	109 min	66 min	54 min
20% class F fly ash	1 MPa	92 min	61 min	43 min
	5 MPa	122 min	78 min	54 min
	13 MPa	147 min	89 min	62 min

the penetration hardness reaches 27.6 MPa is shown in Fig. 7. The casting time (30 min for all) indicates the time need for mixing, casting, and placing inside the microwave chamber. The penetration test was started at the beginning of the heating time to continue until 13 MPa of the penetration hardness, regardless of whether the microwaves were on or off and the specimen was cooling down. The penetration hardness curves were extrapolated from 13 to 27.6 MPa. To reach the final setting time, room temperature cured cement mortars required at least 6 h, but the time was reduced to half of this at 40°C and to only one third of this at 60°C, in Fig. 7. Therefore, the appropriate microwave curing technique enables significant reduction in the time for demolding.

Exploring the possibility of property improvements, appropriate amounts of pozzolanic materials such as blast furnace slag, silica fume, and fly ash were substituted for a portion of the Portland cement and the 28-day compressive strength was determined. As shown in Table 2, the curing times are varied due to the nature of the cementitious materials. Slag and fly-ash blended mortars need longer microwave heating time for the objective hardness levels than cement mortar for all temperature, but silica fume blended mortars needed shorter times. Fig. 8 shows the 28-day compressive strength results of blended mortars. Slag or silica fume raised the strength when specimens are cured at room temperature, but fly ash decreased it. All blended mortars show no degradation of strength when cured at 40°C until the penetration hardness reached 13 MPa. Additionally, adding silica fume raised the strength higher than for room temperature curing. At 80°C curing, strength was decreased greatly regardless of type of pozzolanic material. At 60°C curing, the strength of the specimen heated until 1 MPa is similar to the room temperature cured one, but deceased as heating time increased for the slag and silica fume cases. This trend is less when fly ash is added. Therefore, heating times associated with penetration hardness values of 13 MPa at 40°C or 1 MPa at 60°C are the maximum times for which the 28-day compressive strength is not degraded with or without pozzolanic materials. Because times to reach a

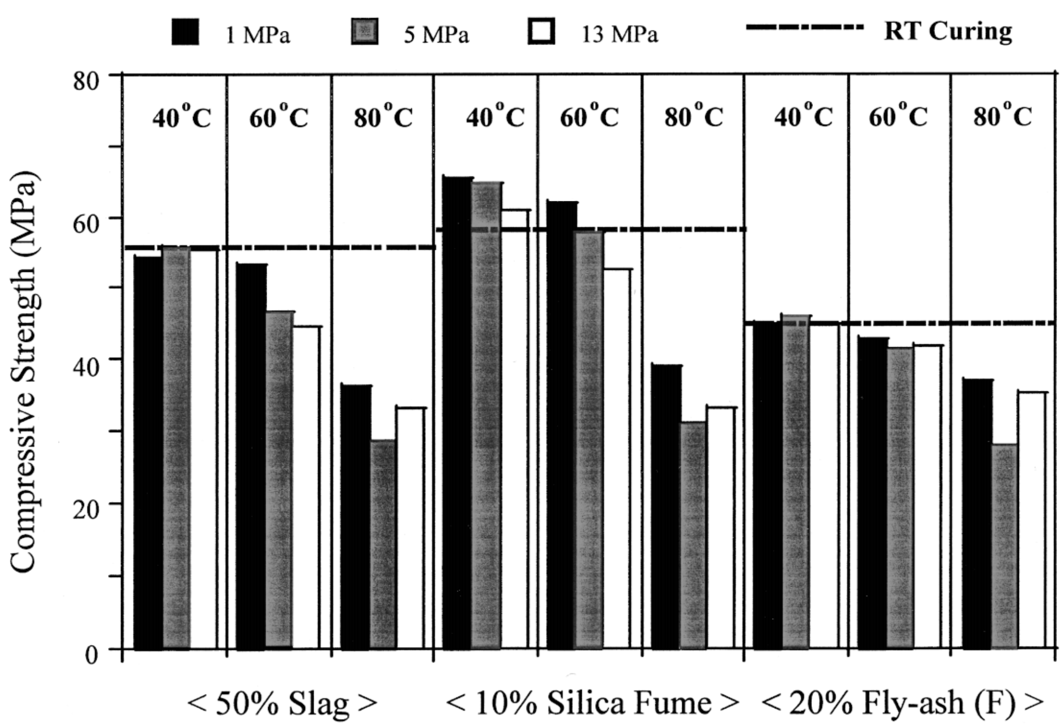

Fig. 8. The 28-day compressive strength for room temperature cured and microwave cured 50% slag, 10% silica fume, and 20% class F fly-ash mortars at 40, 60, and 80°C with sand-to-cement ratio = 2:1 and water-to-cement ratio = 0.4. Specimens were heated until the penetration hardness reached 1, 5, or 13 MPa.

certain penetration hardness are different for each pozzolanic material because of its nature, the penetration hardness test made it possible to monitor the optimum curing time regardless of the amount or type of blended materials.

3. Conclusions

1. Microwave heating with feedback temperature control significantly reduced the final setting times of cementitious mortars without degradation of 28-day strength.
2. Temperature and curing time are important parameters for improvement of 28-day strength of microwave cured cementitious mortars.
3. The optimum conditions were found at 40°C and 60°C for cement and blended mortars, but 80°C was unsuitable for microwave curing.

Acknowledgments

This work was supported by the National Science Foundation through the Science and Technology Center for Advanced Cement-Based Materials under grant no. CHE-91-20002.

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