



THE EFFECT OF NEAT MAGNESIUM OXIDE (MGO) AS EXPANDING ADDITIVE WITH BURNING TEMPERATURE 1200 °C AND 1300 °C ON CEMENT SHEAR BOND STRENGTH AT HIGH PRESSURE AND TEMPERATURE

Aris Buntoro¹⁾
Rudi Rubiandini R.S²⁾.

¹ UPN "Veteran" Yogyakarta;
² Bandung Institute of Technology

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ABSTRACT

Good bonding between cement and casing and between cement and formation is essential for effective zone isolation. Poor bonding will create a problem in production operation both for geothermal wells and oil wells. Expanding additives, such as magnesium oxide (MgO) are used for improving primary cementing. Burning the dolomite (MgCO₃) with high temperature in blast furnace produces the magnesium oxide (MgO) and it is milled with fineness 2800 – 3200 cm²/gram.

The presence of mud film on casing wall and mud cake on formation will decrease shear bond strength, and for the composition without expanding additive it may reach zero. However, by adding expanding additives, shear bond strength of cement will increase, because the improved bonding is the result of mechanical resistance or tightening of the cement against the casing and formation.

The objective of this research is to evaluate the effect of neat magnesium oxide as expanding additive with burning temperature 1200°C and 1300°C on shear bond strength at high pressure and temperature.

1. INTRODUCTION

Bonding between cement and casing is effected by temperature changes, which cause the casing to expand and contract alternately. During the initial setting of the cement slurry, the casing is in an expanded state because of the heat generated from hydration in the cement. Subsequent internal temperature reduction, as the result of mud circulation, can cause the casing to contract and this may destroy the cement/pipe bond partially or wholly.

Expanding additives can alleviate this problem because they tend to expand after the initial set, resulting in the development of a stress condition in the cement that helps to maintain bonding during pressure and temperature changes. The overall result is greatly improved zone isolation. Because the bonding between cement and formation determines its bonding quality, so the increasing of shear bond strength caused by expanding additive become necessary.

This research was performed to evaluate the effect of neat magnesium oxide (MgO) as expanding additive with fineness 2800 - 3200 sqcm/gr and burned at 1200°C and 1300°C on shear bond strength of API Class G Portland cement containing silica flour (SiO₂) 35 %BWOC and various temperatures after 24 hours curing time at 2000 psi^{2,3,6)}. The burning temperature 1200°C and 1300°C was determined based on the previous research²⁾. Laboratory testing performed in modified pressure curing chamber simulator. It was designed to operate at temperature up to 200°C and pressure 2000 psi.

2. LITERATURE REVIEW

Cementing is an integral, necessary aspect of drilling both oil and gas wells and geothermal wells. Cement is used to secure

casing string and to isolate zone for production purpose as well as to solve various hole problems, such as vibration and protect the casing from corrosive fluid attack.

These compounds are formed in a kiln by a series of reactions at temperatures as high as 1500°C between lime, silica, alumina and iron oxide. When hydrated, these compounds make a major contribution to the strength of the cement.

In the manufacturing process selected raw materials are ground to a fine powder, and proportioned in such away that resulting mixture has a desired chemical composition. The raw material consists of two type materials, which are needed to prepare a mixture, i.e. "calcareous" materials, which contain lime, and "argillaceous" materials, which contain alumina, silica and iron oxide. After blending, the raw material mixture is fed into kiln and converted to cement clinker. The clinker is cooled, a small amount of gypsum (3% to 5%) is added, and the mixture is pulverized. The pulverized product is finished Portland cement.

The hydration of Portland cement is a sequence of overlapping chemical reactions between clinker components, calcium sulfate and water, leading to continuos cement slurry thickening and hardening. Although the hydration of C₃S is often used as a model for the hydration of Portland cement, it must be kept in mind that many additional parameters are involved. Portland cement is essentially a calcium silicate material, the most abundant components being tricalcium silicate (C₃S) and dicalcium silicate (C₂S). Upon addition of water, both hydrate to form gelatinous calcium silicate hydrate called "C-S-H gel", which is responsible for the strength and dimensional stability of the set cement at ordinary temperatures. In addition to C-S-H gel, a substantial amount of calcium hydroxide (CH) is liberated.

C-S-H gel is the early hydration product even at elevated temperature and pressure, and is an excellent binding material at well temperatures less than about 230°F (110°C). At higher temperature, C-S-H gel is subject to metamorphosis, which usually result in decreased permeability of the set cement. This phenomenon, known as "strength retrogression", was first reported in the petroleum literature by SWAYZE (1954)⁷ as a result of the growing trend toward deep well completion.

C-S-H gel often converts to the phase called "alpha dicalcium silicate hydrate (α -C₂SH)". α -C₂SH is highly crystalline and much denser than C-S-H gel. As a result, a shrinkage occurs which is deleterious to the integrity of the set cement. Significant loss of compressive strength occurred within one month; however, the level to which strength falls is sufficient to support casing in a well (SUMAN and ELLIS, 1977)⁴. The real problem lies in the severe permeability increases. To prevent interzonal communication, the water permeability of well cements should be no more than 0.1 Md. Within one month, the water permeability of the normal density Class G system (1,2) were 10 to 100 times higher than the recommended limit.

The strength retrogression problem can be prevented by reducing the bulk lime-to-silica ratio (C/S ratio) in the cement (MENZEL, 1935; KALOUSEK, 1952; CARTER and SMITH, 1958)⁴. To accomplish this, the Portland cement is partially replaced by ground quartz, usually as fine silica sand or silica flour. In some areas, special cements are available where quartz has been interground with Portland cement clinker (ITALCEMENTI, 1977; BERRA et al., 1988)⁴. C-S-H gel has a variable C/S ratio, averaging about 1.5. The conversion to α -C₂SH at 230°F (110°C) can be prevented by the addition of 35% to 40% silica (BWOC), reducing the C/S ratio to about 1.0. At this level, a mineral known as tobermorite (C₅S₆H₅) is formed; fortunately, high strength and low permeability are preserved. As the curing temperature increases to about 300°F (150°C), tobermorite normally convert to xonotlite (C₆S₃H) and smaller amount of gyrolite (C₆S₃H₂) with minimal deterioration. Tobermorite sometimes persist to 482°F (250°C) in Portland cement systems because of aluminium substitution in the lattice structure (KALOUSEK and CHOW, 1976)⁴.

Cements containing significant amounts of truscotite are usually characterized by low permeability (GALLUS et al., 1978)⁴. The formation of pectolite, a sodium calcium silicate hydrate, is accompanied by cement expansion (NELSON and EILERS, 1982)⁴; in addition, pectolite appears to render cements more resistant to corrosion by highly saline brines (NELSON and KALOUSEK, 1977; NELSON et al., 1981)⁴. Scawtite has been shown to enhance cement compressive strength when present in minor amounts (EILERS et al., 1983)³. In general, set cements, which consist predominantly of calcium silicate hydrates with C/S, ratios less than or equal to 1.0 tend to have higher compressive strengths and lower water permeability.

The previous researches^{2,3,6} concerns the shear bond strength, which is the adhesion strength between cement and casing and between cement, and formation concluded that the failure of cement was caused by poor bonding. Furthermore, they determined that the shear bond strength minimum was 100 psi after 24 hours curing time. Those researches was also done to understand the compressive strength phenomenon at elevated temperatures by adding silica flour (SiO₂) in varying concentration, and shear bond strength phenomenon, which

never increased at elevated temperatures by adding expanding additives cement, but they tend to expand after the initial set, resulting in the development of a stress condition that helps to maintain the bonding during pressure and temperatures changes.

DANJUSHEVSKY⁵ stated that the increasing of cement volume, it means increasing the relative volume which is affected by the increasing size hardened cement. This increasing volume is caused by :

- **Liquid condition**, is caused by chemical contraction, the other hydrate products is formed such as the formation of salt dissolved crystal at elevated temperature.
- **In curing time condition**, is caused by expanding materials in cement matrix such as CaO, MgO and CaSO₄.
- **After curing time condition**, is caused by salt water in the formations, as the electrolyte charges.

In cementing job, the second point above is an important condition in the increasing of cement shear bond strength.

A result of the DANJUSHEVSKY's research which was done by adding CaO and MgO, where those materials are burned before use as expanding additive at certain temperature, it indicates that the expansion more than 1%, even up to 15% in particular condition.

RUDI RUBIANDINI⁵ in his previous research concerning shear bond strength phenomenon stated that by adding CaO and MgO as expanding additives with the burning temperatures up to 1600°C, and with certain fineness which was tested up to 150°C caused the increasing the shear bond strength varies from 250% to 1000%. He also stated that the silica flour concentration less than 30% BWOC did not improve compressive strength, because the C/S ratio still more than 1.0.

The result of research was performed by ARIS BUNTORO and RUDI RUBIANDINI² conclude that the neat magnesium oxide (MgO) as expanding additive with burning temperature 1400°C and fineness 2000 – 3000 cm²/gram provides the excellent shear bond strength and fair compressive strength of cement in geothermal and oil well at high temperature up to 250°C.

3. LABORATORY TEST

3.1. Simulator Preparation

This research needs specific apparatus, it was a simulator as the modified of pressurized curing chamber which can be operated at temperature up to 350°C with pressure up to 3000 psi. The main parts of the simulator for conditioning cement slurry are :

- Cylindrical simulator is completed with heater and thermocouple in the inside.
- Maximator pump is used to increase pressure from the compressor up to 6500 psig.
- Safety valve or rupture disk
- Reservoir liquid injection reserve
- Temperature regulator
- Flowmeter for Injection gas
- Cooling drum

- Manometer and valves to regulate flow distribution of liquid and gas.

3.2. Curing Molds

For testing both compressive strength and shear bond strength were used three types of curing molds to place cement slurry, these are :

- **First mold**, is a cubic form with the size $2 \times 2 \times 2$ in. This mold is used to test API standard compressive strength.
- **Second mold**, is a cylindrical form with the size inside diameter and height are 1 inch and 2 inches respectively, and it is also completed with the cap both at the top and the bottom. This mold is used to test shear bond strength between cement and casing.
- **Third mold**, is a cylindrical core with the hole diameter 1 inch and height 1.5 inches. This mold consists of six holes to place the cement slurry. The specimens from this mold are used to test compressive strength, and cement permeability without casing.

These molds were designed to place into the simulator simultaneously before testing.

3.3. Specimens Test

Using API Class G Portland cement - HSR Type, with the following composition did this test:

- Water Solids Ratio (WSR) = 44%
- Silica Flour (SiO_2) = 35% BWOC
- Neat Magnesium oxide (MgO) varies from 0 to 10% BWOC

As the medium of curing chamber was used fresh water and the duration of test was designed for 24 hours (1 day).

Calculation of Compressive Strength

The set specimens of cement are removed from the molds, then placed in a hydraulic press where increasing uniaxial pressure is exerted on each until failure. The compressive strength is then calculated from the pressure at which failure occurred by cross-sectional area of specimen. Using the equation below can do the calculation of compressive strength

$$CS = kP \left(\frac{A_1}{A_2} \right) \quad (1)$$

where CS is compressive strength, psi ; P is maximum load, psi ; A_1 and A_2 are block bearing cross section area at the hydraulics mortar, $sq\text{in}$ and specimen cross section area, $sq\text{in}$; k is correction constant, is a function of the height/ diameter (t/d) ratio.

Calculation of Shear Bond Strength

In this test assumed that casing is real clean. The shear bond strength test is done same as the compressive strength test, but with a driving stick and cylindrical holding. The pressure at which initial shear occurred, is the maximum shear stress. Shear bond strength can be calculated by using the equation below :

$$SBS = P \left(\frac{A_1}{\pi d h} \right) \quad (2)$$

where SBS is shear bond strength, psi ; P is maximum shear stress, psi ; A is block bearing area at the hydraulic mortar, $sq\text{in}$; d is inside diameter of casing, in ; h is the height of specimen in cylindrical mold, in.

4. RESULT OF THE LABORATORY TEST AND DISCUSSION

4.1. Result of the Laboratory Test

The results of the laboratory test were taken from the adding of neat MgO with burning temperature $1200^{\circ}C$ and $1300^{\circ}C$ with fineness 2800 - 3200 cm^2/gram at various concentration from 3, 5, 7, and 10% as shown in **Figures-1 to 6**.

Figures-1, 2 and 3 are the diagrams depicting the effect of neat magnesium oxide (MgO) with burning temperature $1200^{\circ}C$ as expanding additive on shear bond strength (clean casing), shear bond strength (with mud cake), and compressive strength respectively at temperatures vary from $100^{\circ}C$, $135^{\circ}C$, $150^{\circ}C$ and $200^{\circ}C$; at constant pressure (2000 psi); and the duration of curing time is 24 hours (1 day). **Figures-4, 5 and 6** are the diagrams depicting the effect of neat magnesium oxide (MgO) with burning temperature $1300^{\circ}C$ as expanding additive on shear bond strength (clean casing), shear bond strength (with mud cake), and compressive strength respectively at temperatures vary from $100^{\circ}C$, $135^{\circ}C$, $150^{\circ}C$ and $200^{\circ}C$; at constant pressure (2000 psi); and the duration of curing time is 24 hours (1 day).

4.2. The Effect of Temperature

The Adding Neat Magnesium Oxide (MgO) with Burning Temperature $1200^{\circ}C$

Figure 1 to 3 indicates that the addition of 10% BWOC neat magnesium oxide at temperature $100^{\circ}C$ provides the increasing shear bond strength significantly. However, the presence of mud film on the casing wall will decrease shear bond strength. It can be seen that the cement specimen without magnesium oxide, its shear bond strength value decrease from 479 psi to 91 psi, but with addition of magnesium oxide, the shear bond strength value increase sharply, i.e. from 91 psi to 255 psi. Meanwhile, its compressive strength value decreases slightly, i.e. from 4422 psi to 3957 psi.

At temperature $135^{\circ}C$, there is an interesting phenomenon, because the decrease of compressive strength values at the whole MgO concentration. Even so, their values still higher than minimum value according to API Spec, i.e. 1000 psi.

Addition 10% BWOC of neat magnesite at temperature $150^{\circ}C$ indicates that shear bond strength increase sharply, i.e. from 864 psi to 2319 psi, meanwhile, with the presence of mud film on the casing wall, the shear bond strength without magnesium oxide decrease up to 121 psi, but with the adding of MgO the shear bond strength value increase until 3 times.

Finally, the addition of MgO at temperature $200^{\circ}C$ shows that 7% BWOC of neat magnesium oxide increase the shear bond strength value sharply, meanwhile its compressive strength value decrease slightly. And, the presence of mud film on the casing wall, the cement specimen without MgO decrease up to 155 psi, but with the adding of MgO the value of shear bond

strength increase to 783 psi (7% BWOC of MgO) and 811 psi (10% BWOC of MgO).

The Adding Neat Magnesium Oxide (MgO) with Burning Temperature 1300°C

Figure-4 to 6 indicates that the addition of 5% BWOC neat magnesium oxide at temperature 100°C provides the maximum shear bond strength. However, the presence of mud film on the casing wall will decrease shear bond strength. It can be seen that the cement specimen without magnesium oxide, its shear bond strength value decrease from 479 psi to 91 psi, but with addition of magnesium oxide, the shear bond strength value increase sharply, i.e. from 91 psi to 311 psi (7% BWOC of MgO), and, its compressive strength value decrease.

At temperatures 135°C, 150°C and 200°C shows that concentration 5% provides the highest compressive strength at temperature 150°C, i.e. 4518 psi. Shear bond strength value at 5%BWOC neat MgO also provides the highest from 100°C to 150°C, then it decreases slightly at 200°C. And, the presence of mud film on the casing wall will decreases the shear bond strength value for whole concentration.

Basically, the MgO with burning temperature 1300°C at concentration 3%, 5%, and 10% can be used excellently at temperature 150°C and 200°C.

5. CONCLUSIONS

We have reached the following conclusions from this laboratory study on the effect of neat magnesium oxide (MgO) as an expanding additive on cement shear bond strength at high temperature up to 200°C :

1. Magnesium oxide with burning temperature 1200°C has excellent perform at 150°C with concentration 10%BWOC on shear bond strength value, where it increases until 300%. Meanwhile, its compressive strength values still higher than minimum value according to API Spec (1000 psi).
2. Magnesium oxide with burning temperature 1200°C (7%BWOC) at 200°C gives the maximum shear bond strength (2718 psi).
3. Magnesium oxide with burning temperature 1300°C (7%BWOC) at 200°C also provides the maximum shear bond strength (2102 psi).
4. Both magnesium oxides with burning temperature 1200°C and 1300°C increases shear bond strength vary from

250% to 300%, and it can be used as expanding additive to overcome the poor bonding problem in geothermal and oil wells, especially at elevated temperature.

5. The presence of mud film on the casing wall will decreases shear bond value until 500%. Thus, it needs the adding of neat MgO as expanding additive to alleviate this problem.

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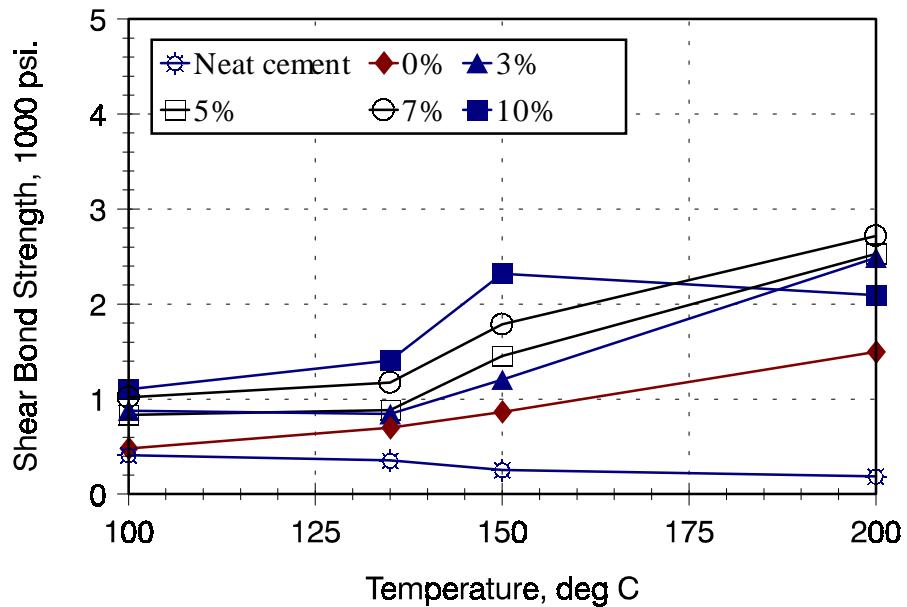


Figure-1
Effect of neat MgO with burning temperature 1200°C on the shear bond strength of API Class G Portland cement containing silica flour (SiO₂) 35 %BWOC and various temperatures after 24 hours curing time at 2000 psi

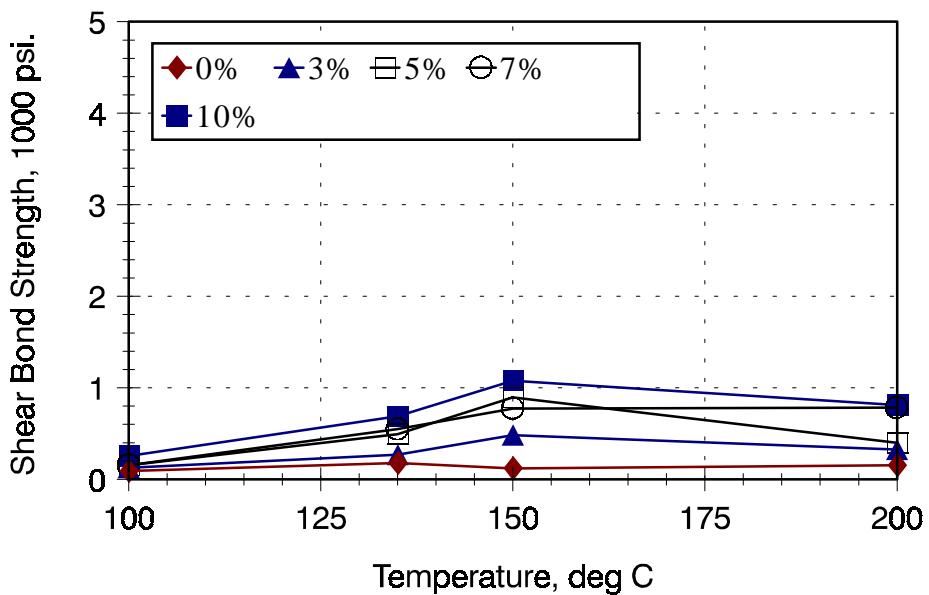


Figure-2
Effect of neat MgO with burning temperature 1200°C on the shear bond strength of API Class G Portland cement containing silica flour (SiO₂) 35 %BWOC and various temperatures after 24 hours curing time at 2000 psi (with mud cake)

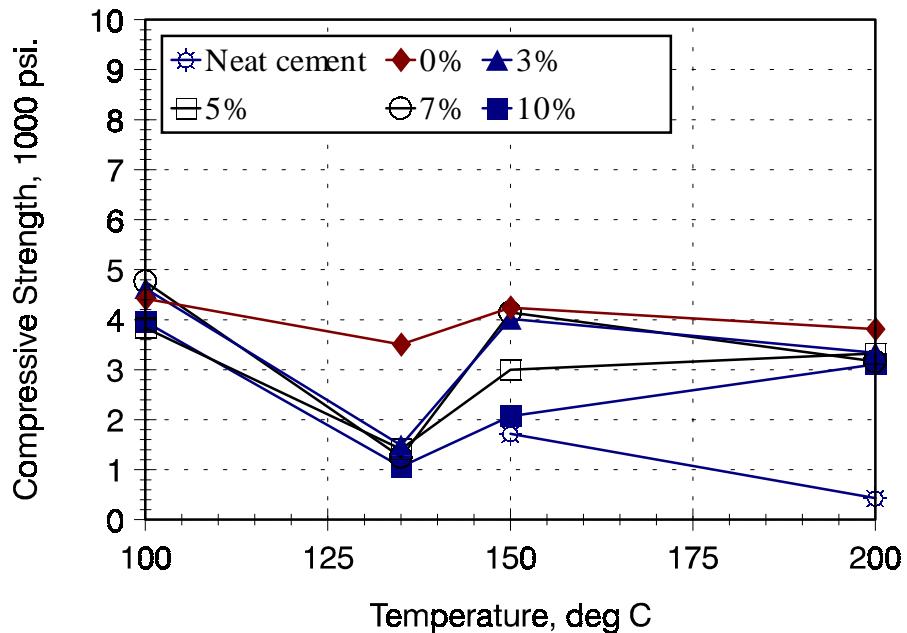


Figure-3
Effect of neat MgO with burning temperature 1200°C on the compressive strength of API Class G Portland cement containing silica flour (SiO₂) 35 %BWOC and various temperatures after 24 hours curing time at 2000 psi

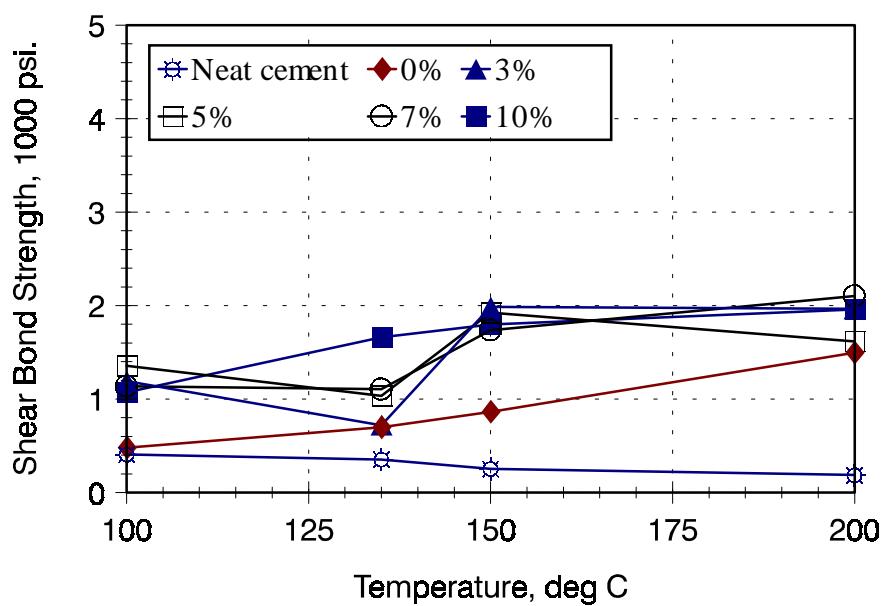


Figure-4
Effect of neat MgO with burning temperature 1300°C on the shear bond strength of API Class G Portland cement containing silica flour (SiO₂) 35 %BWOC and various temperatures after 24 hours curing time at 2000 psi

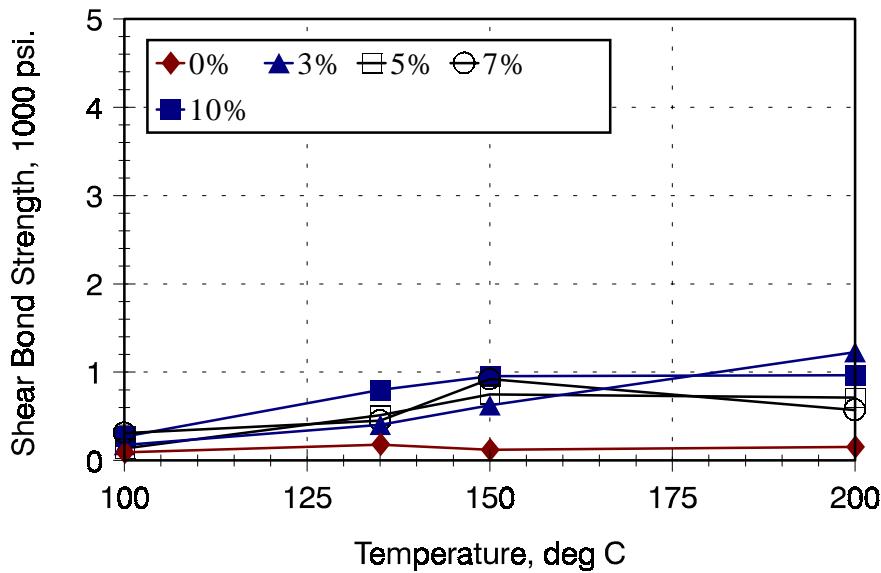


Figure-5
Effect of neat MgO with burning temperature 1300°C on the shear bond strength of API Class G Portland cement containing silica flour (SiO₂) 35 %BWOC and various temperatures after 24 hours curing time at 2000 psi (with mud cake)

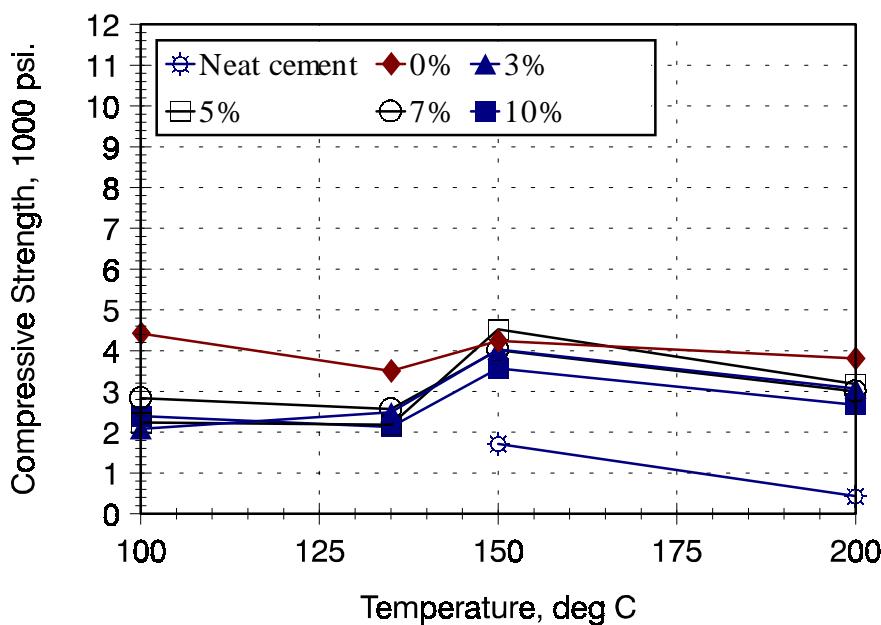


Figure-6
Effect of neat MgO with burning temperature 1300°C on the compressive strength of API Class G Portland cement containing silica flour (SiO₂) 35 %BWOC and various temperatures after 24 hours curing time at 2000 psi