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ASSISTANCE IN LABORATORY SCALE INVESTIGATION ON MAGNESITE ORE AND MAGNESITE BRICKS

UC/ZIM/85/199
ZIMBABWE

Final Report

PREPARED FOR THE GOVERNMENT OF ZIMBABWE BY THE UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION

Based on the work of Research Institute MAGNOHROM, Kraljevo, YUGOSLAVIA

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ASSISTANCE IN LABORATORY SCALE INVESTIGATION ON MAGNESITE ORE AND MAGNESITE BRICKS INVESTIGATION ON THE POSSIBILITY OF OBTAINING HIGH QUALITY DEAD BURNED MAGNESITE, MAGNESITE TEST BRICKS AND MAGNESITE-CHROME TEST BRICKS OF UPGRADED CALCINED MAGNESITE FROM ZIMBABWE

UC/ZIM/84/199
ZIMBABWE

Prepared for the Government of Zimbabwe
by the United Nations Industrial Development Organisation,
executing agency for the United Nations Development Programme

Based on the work of Research Institute,
MAGNOHROM, Kraljevo, YUGOSLAVIA

United Nations Industrial Development Organization
Vienna

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Final Report

-1695-

Prepared for the Government of Zimbabwe by the United Nations Industrial Development Organization, executing agency for the United Nations Development Programme

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A B S T R A C T

Direct usage of Zimbabwean magnesite, due to high CaO content, is rather limited. Therefore, in 1983, investigations on Kadoma magnesite ore beneficiation were finished and reduction of CaO content of over 50% was achieved.

These investigations are natural continuation of events. They include: Possibility of obtaining high grade dead burned magnesite, magnesite bricks and magnesite-chrome bricks of beneficiated calcined Zimbabwean magnesite.

Initial material for the investigations were calcined unrated magensites SP-A and SP-B, produced by selective hydration procedure in Zimbabwean institutes.

On the semi-product SP-A additional reduction of CaO content was achieved by screening on a 3 mm mesh screen.

Starting with semi-products SP-A and SP-B and applying activities of milling, briquetting and dead burning, dead burned magnesites DBM-A and DBM-B were produced.

Dead burned magnesite DBM-A with 95-96% of MgO ranges to a high grade materials, and DBM-B, with 91-92% of MgO ragnes to middle grade of dead burned magnesites, obtained of natural raw materials.

Of these dead burned magnesites, test magnesite bricks and magnesite-chrome bricks were produced, with good characteristics. The obtaind grades of these bricks is comparable to corresponding grades which are successfully used in heating units for production of steel, copper and cement.

The wollowing was recommanded:

1. To start with semi-industrial investigations as soon as possible, in order to:
   - confirm the results obtained on laboratory scale;
   - provide technological parameters needed for elaboration of feasibility study;
   - produce adequate quantity of brick which would be tested by potential consumers in Africa.
2. To reconstruct the existing semi-industrial rotary kiln, 11 m of length, at direct vicinity of Kadoma magnesite mine.
3. To perform semi-industrial investigations of calcining and beneficiation in Zimbabwe, and others in Yugoslavia/Europe.

Contents:

I. CONCLUSIONS AND RECOMMANDATIONS

A. Conclusions

1. The semi-products SP-A and SP-B, as obtained from upgrading calcined magnesite by selective hydration method in Zimbabwean institutes, were used for these investigations. For the detaills kindly consult UNIDO-Report No. DP/ZIM/83/006, March 1985.

   Additional screening of the semi-product SP-A (2.36 - 16 mm) on 3 x 3 mm screen proved useful. The improved semi-product SP-A´ (3 - 16 mm), with CaO content below 3 %, was obtained. Reduction of lime content will enable use of semi-product SP-A´ for production of higher quality dead burned magnesite, as well as magnesite bricks and magnesite-chrome bricks.

2. To the semi-product SP-B (1.18 - 2.36 mm) fraction 2.36 - 3.0 mm, as sieved out from the original semi-product SP-A, was added. Thus, the semi-product SP-B´ (1.18 - 3 mm), now having lower lime content (4.42 %), was obtained. This material, with some additives, can be used for production of normal quality dead burned magnesite and magnesite bricks.

3. Applying the proposed method of beneficiation of raw magnesite from Kadoma (calcining, selective hydration, and screening on 1.18 mm and 3 mm screen, respectively) the following recoveries of the semi-products and their lime contents were achieved.

Semi-product	Fraction, mm	Recovery	Lime content, %
SP-A´	-16 +3	35-40	2.5 - 3
SP-B´	-3 +1.18	30-35	4 - 5
Tailings	-1.18	28-32	10 - 15
Input	-25 +0	100	6.75

So, the total recovery of both semi-products, SP-A´ and SP-B´, amounting up to about 70%, which can be considered as very good for breccia-type raw magnesite having high lime content, was achieved.

4. By direct dead burning of semi-products SP-A´ under stationary conditions at the temperature of 1800°C, dead burned magnesite, designated as DBM-AD, consisting of light and dark gray grains, was obtained. Within mineralogical composition of light grains, beside periclase, the following silicates were found: forsterite ($2MgO.SiO_2$), monticellite ($CaO.MgO.SiO_2$), merwinite ($3CaO.MgO.2SiO_2$) and dicalcium silicate ($2CaO.SiO_2$). In dark gray grains, in addition to periclase, prevailingly dicalcium silicate and some free lime were found.

With over 95% of MgO and bulk density of 3.31 g/cm^3, this dead burned magnesite ranges to the highest quality ones, produced of natural magnesites. But, the main disadvantage of this product is its rather inhomogeneous composition and presence of free lime, causing low hydration resistence.

5. By additional processing of semi-product SP-A´, trough fine milling, briquetting and dead burning, dead burned magnesite, designed as DBM-A, was obtained.

Dead burning was performed under stationary conditions at temperatures of 1700, 1800 and 1900°C. Increased temperature of dead burning brought about better characteristics of dead burned products. At the temperature of 1800°C, obtained dead burned magnesite had bulk density of 3.32 g/cm^3 and average size of periclase crystals of about 70 µm. Chemical and mineralogical compositions of this dead burned magnesite were homogeneous, MgO content high (96.13%) and free lime content very low (0.15%). Mineralogicaly, this dead burned magnesite consists prevailingly of periclase (MgO) and of dicalcium silicate ($2CaO.SiO_2$) and tricalcium silicate ($3CaO.SiO_2$). With MgO content of over 95%, bulk density of over 3.30 g/cm^3 and very good hydration resistance, the dead burned magnesite DBM-A belongs to the highest grades, produced of natural magnesites.

6. Additional processing of semi-product SP-B' was achieved by fine milling, by additions of iron oxide or SiO_2, by briquetting and by dead burning. Dead burned magnesite obtained with addition of iron oxide was designated as DBM-BF and the other one, with addition of SiO_2, as DBM-BS.

The obtained dead burned magnesite, DBM-BF, has shown homogeneous chemical and mineralogical compositions, with MgO content of about 91%. Prericlase (MgO) is the main constituent of its mineralogical composition, and then come dicalcium silicate ($2CaO.SiO_2$), tricalcium silicate ($3CaO.SiO_2$) and dicalcium ferrite ($2CaO.Fe_2O_3$). Hydration resistance is very good.

By dead burning at 1800°C mean size of periclase crystals of about 100 μm was obtained, as well as apparent density of 3.27 g/cm^3. By finer milling and by applying higher specific pressure during briquetting, obtaining of apparent density of 3.30 g/cm^3 is expected.

With these characteristics, this dead burned magnesite ranges to medium grades, produced of natural magnesites.

Dead burned magnesite DBM-BS, produced with addition of SiO_2, has the content of MgO of 92.37% and beside basic mass of periclase, dicalcium silicate is also present. Its hydration resistance is satisfactory.

By dead burning at 1800°C mean size of periclase crystals of about 70 μm and apparent density of 3.25 g/cm^3 were obtained.

By finer milling and by applying higher specific pressure during briquetting, obtaining apparent density of 3.30 g/cm^3 is expected.

With these characteristics this dead burned magnesite belongs to medium grades, produced of natural magnesites.

7. On basis of dead burned magnesite DBM-A, magnesite bricks were produced and designated as M-A, with MgO content of 95.62%. Mineralogical composition of these bricks consists of periclase (main constituent), dicalcium silicate and tricalcium silicate. Ceramic bond is direct (periclase-periclase) or achieved by means of highly refractory silicates, i.e. dicalcium and tricalcium silicate. The bricks are of the following characteristics: low porosity, good apparent density, satisfactory cold crushing strength, high refractoriness under load and very good thermal shock resistance.

   On basis of the results obtained for chemical and mineralogical compositions, microstructural characteristics and physical properties, it can be concluded that magnesite brick M-A belongs to the highest grade of this type, made of natural magnesites.

8. Magnesite bricks designated with M-BF, having 92.1% of MgO, were produced of dead burned magnesite DBM-BF. Their mineralogical composition consisted of: periclase (main constituent), dicalcium silicate, tricalcium silicate and dicalcium ferrite. These bricks have shown low porosity, high apparent density, good cold crushing strength and excellent thermal shock resistance. Refractoriness under load is also very good, though some dicalcium ferrite is present. This is due to predomimant effect of highly refractory phases, i.e. periclase, dicalcium silicate and tricalcium silicate. On basis of the results obtained, the bricks M-BF belong to medium grade magnesite bricks.

9. On basis of dead burned magnesite DBM-BS, magnesite bricks designated with M-BS, were produced, which contained 91.85% of MgO. Beside the main constituent, periclase, only the presence of dicalcium silicate was observed. Microstructure of this brick shows that ceramic bond is either direct (periclase-periclase), or is achieved by means of dicalcium silicate. The bricks are of somewhat higher porosity and lower apparent density. Cold crushing strength is satisfactory. Very good refractoriness under load was achieved.

By adequate improvement of brick mixture composition, lower porosity values, within usual limits (16-20%), and apparent density of 2.85 - 3g/cm^3, could be achieved.

On basis of the results obtained, it can be concluded that the bricks MB-S belong to medium grade magnesite bricks.

10. Taking the obtained results in consideration, it could be said that in magnesite bricks M-BF and M-BS, produced of corresponding qualities of dead burned magnesites, i.e. DBM-BF and DBM-BS, high level of free lime "stabilization" was achieved.

    However, both methods of "stabilization", either by adding Fe_2O_3, or by adding SiO_2, have their advantages or disadvantages, as far as effect on bricks properties is concerned. It was concluded that the best results could be achieved with mixture of both additives, in order to chemically bind free lime during dead burning process.

11. For production of magnesite-chrome bricks designated with MC-A, the following starting materials were used:

    - Dead burned magnesite DBM-A
    - Lumpy chrome ore from Frances Mine, Zimbabwe (Representative sample taken in July 1982) and
    - Chrome ore concentrate from Mtoroshanga Mine, Zimbabwe (Representative sample taken in July 1982).

    Content of Cr_2O_3 in these bricks was 16.97%.

    Microstructure of these bricks contained the following mineral phases: periclase, chromite, secondary chrome spinellides and dicalcium silicate. Chemical bond was based either on direct contact between periclase and chromite or on dicalcium silicate. The bricks had a satisfactory cold crushing strength, good thermophysical properties, low porosity, good apparent density and good thermal shock resistance.

    On basis of the results obtained for chemical and mineralogical compositions, structural characteristics and thermophysical properties, it can be concluded that the obtained magnesite--chrome bricks MC-A are of very good quality.

12. Generaly speaking, the resultas of laboratory scale investigations on the possibility of obtaining high quality dead burned magnesite, magnesite bricks and magnesite-chrome bricks of upgraded local magnesite, chrome ore and chrome ore concentrates, are very positive.

The obtained dead burned magnesite DBM-A, with properties of highest quality products, that can be produced of natural magnesites, can be used for production of magnesite bricks, magnesite-carbon bricks and magnesite-chrome bricks, belonging also to the highest grade basic bricks.

The obtained magnesite test bricks and magnesite-chrome test bricks, designates as: M-A, M-BF, M-BS and MC-A, according to their characteristics can compete with corresponding brick grades usually used for lining of industrial kilns and furnaces in steelmaking, copper production, cement industry, and the like.

Dead burned magnesite DBM-A, as well as magnesite bricks M-A, and mahnesite-chrome bricks MC-A, by their characteristics range to the highest grade of basic refractories produced of natural raw materials (magnesites).

B. <u>Recommandations</u>

1. It is recommanded that carrying out of semi-industrial investigations be done as soon as possible, as on laboratory scale basic refractory products were obtained ranging by their properties to the highest grade basic refractories produced of natural magnesites. The main aim of the proposed semi-industrial investigations is to avoid the risk of an unpleasant surpeise of any kind and to get more reliable results for evaluation of products quality in all phases of the production process.

This is suggested because conditions under which semi-industrial investigations are to be carried out are much more similar to those in actual industrial production. Process of calcining, and particularly of dead burning, ought to be done under dynamic conditions, which is even more favourable for products quality.

Another important aim of semi-industrial investigations is to provide relevant parameters, such as: specific consumption of raw materials, additives, energy, etc., needed for FEASIBILITY STUDY elaboration.

The last, but not the least important aim of semi-industrial investigations is to produce about 10-12 tonnes of basic refractory bricks, which would be actually tested by their installment in industrial kilns and furnaces at potential consumers plants in Africa.

Detailled Program of semi-industrial investigations is attached to this Report as Annex No. 2.

2. It is recommanded to carry out the proposed semi-industrial investigations in Zimbabwe to the greatest possible extent, depending mostly on the existing equipment and on equipment listed in Annex 3, that can be easily purchased with no much investment. Procurement of the equipment ought to be done in due time, i.e. before beginning of investigations.

The first part of investigations, which is to be carried out in Zimbabwe, comprises the following:

- Collecting of representative samples of raw magnesite, chrome ore, chrome ore concentrate and flake graphite;
- Determination of the characteristics of all representative samples;
- Calcining of raw magnesite, and
- Upgrading of calcined material by the method of selective hydration.

The second part of investigations should be carried out in Yugoslavia/Europe, where it can be done more competently, economically and in shorter time period. It should be born in mind that 10-12 tonns of refractory bricks of highest grade are going to be produced.

3. It is recommanded to adapt the existing semi-industrial rotary kiln, 10400 mm of lenght, in Eiffel Flats, near Kadoma, Zimbabwe, and to prepare all other needed equipment for execution of the proposed semi-industrial investigations. This also to be done before beginning of work.

4. It is recommanded that Government of Zimbabwe should further explore market in the countries of SADCC and PTA and collect additional information necessary for preparation of MARKETING ANALYSIS for basic refractories.

5. It is recommanded to GEOLOGOCAL SURVEY, Harare, to check and confirm reserves of raw magnesite at Kadoma, with maximum of 1% of silica and 4% of lime.

6. It is recommanded to DEPARTMENT OF METALLURGY to employ some new personnel to be trained as soon as possible as members of counterpart team for work on this project.

7. It is recommanded to start with preparations for elaboration of the FEASIBILITY STUDY for the potential basic refractories plant in Zimbabwe, providing that:

   - semi-industrial investigations solve successfully all technological problems;
   - MARKETING ANALYSIS confirms the sale of 20,000 - 25,000 tonns of basic refractories per annum was ensured;
   - GEOLOGICAL SURVEY, Harare, confirms existence of sufficient magnesite ore reservs at Kadoma, meeting demands of potential basic refractories plant for 20-year operation.

8. It is recommanded to start with designing and preparing the detailled plans and drawings for the basic refractories plant

in Zimbabwe, after the FEASIBILITY STUDY had proved the project economical.

9. It is recommanded to start the training of technical personnel, during construction of the plant, in order to have them competent for work at this new basic refractories plant.

10. It is recommanded to assign sufficient number of experts from the know-how supplier for setting to work the potential basic refractories plant. The experts should stay in Zimbabwe for extended time period, as supervisors and instructors, also taking care of personnel training on site.

II. INTRODUCTION

As there are big reserves of refractory chrome ore and raw magnesite in Zimbabwe, one of primary tasks has been to develope refractories industry in order to evoid import of refractories. The starting basis should be production of dead burned magnesite of quality suitable for the production of all kinds of refractories.

At the requiestof Zimbabwean Government first help on realization of this task has been given by Yougoslav Government, and, later, by UNIDO. In this respect, preliminary study was made (1983) which proved that avalilable chrome ore was suitable for production of all kinds of basic refractories, while raw magnesite could be used only for production of iferiour grades. It was recommanded to continue investigations in order to discover methods of beneficiation of local magensite by reduction of high CaO content and enable production of all qualities of basic refractories.

With this aim extensive laboratory investigations were performed in Zimbabwean institutes. At the begining of March 1985 the Technical Report was submited to UNIDO, the title of which was

> INVESTIGATIONS ON THE POSSIBILITY OF BENEFICATING OF LOCAL RAW MAGENSITE FOR THE PRODUCTION OF BASIC REFRACTORIES (DP/ZIM/83/006).

For these investigations, representative sample of magnesite from regular production of Kadoma Mine, was used. The sample was of the following chemical composition:

Loss on ignition	50.23 %
SiO_2	0.60 %
Al_2O_3	0.13 %
Fe_2O_3	0.12 %
CaO	3.40 %
MgO	44.63 %
Grain size distribution . . .	3 - 25 mm.

The results of investigations in Zimbabwean institutes confirmed that new method has been found as an innovation in the field of beneficiatoion of raw breccia type magnesites with high CaO content, by which reduction of CaO content for over 50% was achieved and the following products obtained:
a) semi-product "A" with 3.26% of CaO; b) semi-product "B" with 5.47% of CaO and c) fraction with over 13% of CaO.

The applied method of magnesite beneficiation consisted of precalcining, controlled wetting and soaking and, finally, screening on adequate sieves. The method is very simple; it uses heat energy developed during the process itself and does not require qualified manpower. It also has comparatively low processing and investment costs.

The upgraded product is expected to, as semi-product "A", enable production of 95% MgO dead burned magnesite and as semi-product "B", production of dead burned magnesite of normal quality. This was to be checked by second phase of laboratory investigations.

Due to lack of adequate equipment in Zimbabwean institutes, the second phase continued in Magnohrom Institute in Kraljevo. These investigations included:
- additional determination of characteristics of the semi-products "A" and "B";
- production of "A" and "B" grades of dead burned magnesite with determination of their characteristics, and

- production of magnesite and magnesite-chrome bricks with determination of their characteristics.

Detailed program of the investigations performed at the Institute in Kraljevo is enclosed with this Report, as Annexe No 1. Realization of all previous investigations on this project was enabled by participations of the following partners:

- UNIDO (UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION), Vienna
- MINISTRY OF MINES, Harare, Zimbabwe
- SOLIDARITY FUND OF FEDERAL EXECUTIVE COUNCIL, Beograd
- REPUBLIC DEPARTMENT FOR INTERNATIONAL SCIENTIFIC, EDUCATIONAL-CULTURAL AND TECHNICAL COOPERATION OF SERBIA, Beograd
- YUGOSLAV BANK FOR INTERNATIONAL ECONOMIC COOPERATION, Beograd and
- MAGNOHROM, Kraljevo.

III. CHARACTERISTICS DETERMINATION ON CALCINED BENEFICIATED MAGNESITE

The samples of calcined beneficiated magnesite of semi-products "A" and "B", produced by procedure of selective hydration in Zimbabwean institutes, were transported to Magnohrom Institute at Kraljevo for further investigations. Designations and quantities are given on table 1.

Table 1.
Details of semi-products samples

Designation	Class	Quantity
SP-A	-16+2.36 mm	120 kgs
SP-B	-2.36+1.18 mm	51 kgs

A. Characteristics of delivered samples SP-A and SP-B

On these samples grain size distribution and chemical composition were determined, and the results obtained are given on tables 2 and 3.

Table 2.
Sieve analysis of samples SP-A and SP-B

Class (mm)	SP-A (%)	SP-B (%)
+16	2.52	
-16 +10	9.03	
-10 +5	33.19	
-5 +3	26.06	
-3 +2	17.50	8.20
-2 +1	6.70	68.40
-1	5.00	23.40

Table 3.

Chemical composition of received samples SP-A and SP-B

Sample design.	Class (mm)	Ign. loss	SiO_2 %	Al_2O_3 %	Fe_2O_3 %	CaO %	MgO %
SP-A	-16+2.36	5.88	0.70	0.14	0.15	3.16	89.97
SP-B	-2.36+1.18	6.51	0.65	0.12	0.14	4.56	88.02

The results of grain size distribution of the SP-A sample show that after three months of storage and by transportation and by handling, some additional comminution occured. To check the CaO content of various classes, chemical analyses were done and the results obtained presented at table 4.

Table 4.

Chemical composition of the SP-A sample per classes in %

Sample design.	Class (mm)	Ign. loss	SiO_2 %	Al_2O_3 %	Fe_2O_3 %	CaO %	MgO %
	16 + 3	5.58	0.63	0.10	0.16	2.84	90.24
SP-A	-3+2	6.50	0.72	0.14	0.13	3.25	89.26
	-2	7.10	0.85	0.15	0.12	4.04	87.74

Chemical composition found in the SP-A sample per classes shows that by screeneng on 3 mm sieve the -16+3mm class can be obtained with CaO content under 3%.

The 2.36-3 mm class contains 3-4% of CaO and therefore can be added to semi-product SP-B and improve its quality.

B. Characteristics of the samples SP-A´ and SP-B´ obtained after screening on 3 mm sieve

Samples of semi-product after screening on 3 mm sieve were marked with SP-A´ and SP-B´. On these samples the following determinations were made: grain size distribution (Table 5), chemical composition (Table 6), mineralogical composition and differential thermal analysis (DTA).

Table 5.
Sieve analysis of samples SP-A´ and SP-B´

Class (mm)	SP-A´ (%)	SP-B´ (%)
+16	0.54	
-16+10	9.00	
-10+5	48.00	
-5+3	35.46	
-3+2	7.00	17.96
-2+1		66.38
-1		15.66

Table 6.
Chemical composition of the samples SP-A´ and SP-B´

Samples	Class (mm)	Ign. loss	SiO_2 %	Al_2O_3 %	Fe_2O_3 %	CaO %	MgO %	B_2O_3 %
SP-A´	-16+3	5.94	0.63	0.10	0.16	2.84	90.33	0.016
SP-B´	-3+1.18	6.61	0.87	0.12	0.18	4.42	87.80	0.02

Mineralogical composition of semi-products SP-A´ and SP-B´

Mineralogical composition was determined on X-Ray diffractometer. On X-Ray patterns, figures 1 and 2, the following minerals were registered (Table 7):

Table 7.

Mineralogical composition of samples

SP-A´	SP-B´
Periclase	Periclase
Brucite	Brucite
Portlantide	Portlantide
Calcium silicates	Calcium silicates
	CaO

These investigations show that the semi-products obtained consist mainly of periclase (MgO), that during selective hydration process CaO mainly changed into Portlantide ($Ca(OH)_2$), and that some of MgO changed into Brucite ($Mg(OH)_2$). In SP-B´ product there was some bigger quantity of Portlantide.

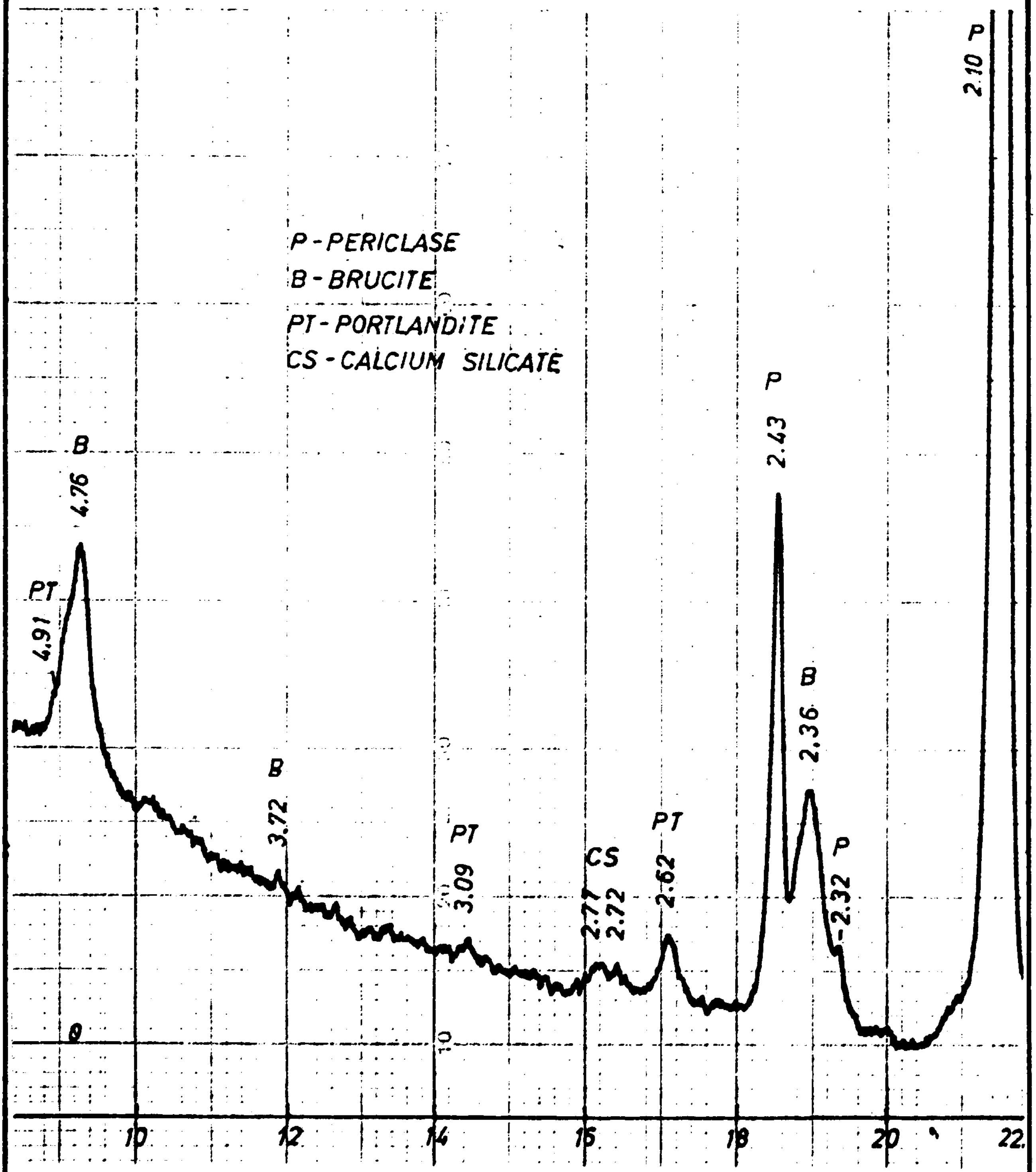

Figure 1. X-Ray pattern of semi-product SP-A´

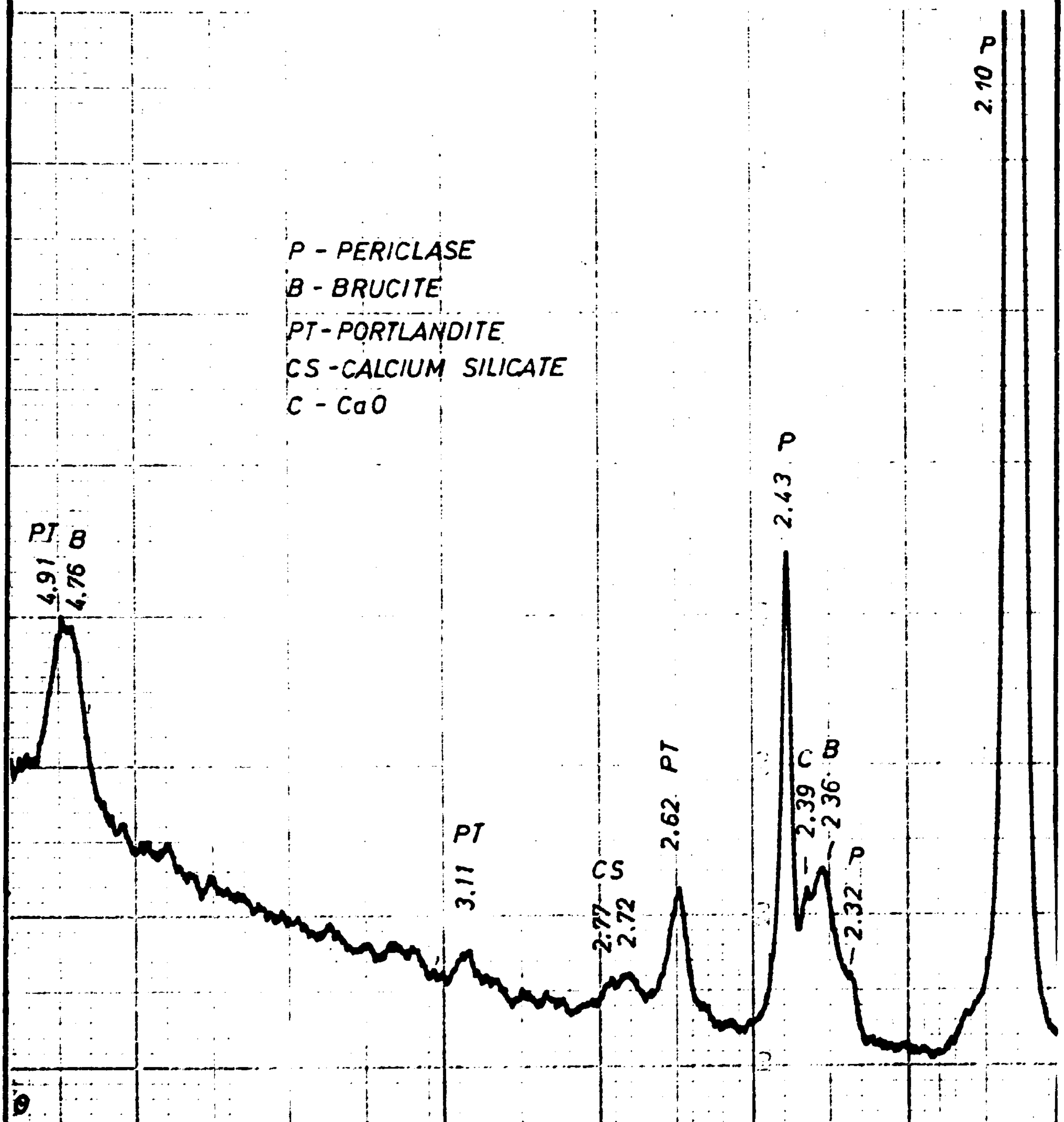

Figure 2. X-Ray pattern of semi-product SP-B´

Differential thermal analyses

In order to determine changes on semi-products A´ and B´ that occur on heating to 1000°C, differential thermal analyses were done. Aparatus of Gebrüder Netsch, having capacity of 10 K/min and maximum sensitivity of differential galvanometer of 0.06 mV/mm, was used. Investigations were done in air. α-Al_2O_3 was used as standard sample. On the thermogram (figure 3) differential thermal curves with two endothermic peaks are given.

The first one, on 380°C, corresponds to dissociation of:

$$Mg(OH)_2 \longrightarrow MgO + H_2O.$$

The other one, at 480°C, corresponds to dissociation of:

$$Ca(OH)_2 \longrightarrow CaO + H_2O.$$

The peaks corresponding to dissociation of $Mg(OH)_2$ are of similar intensity, while the peak corresponding to dissociation of $Ca(OH)_2$ is considerably bigger for semi-product B´. This is in accordance with chemical and mineralogical compositions of products A´ and B´.

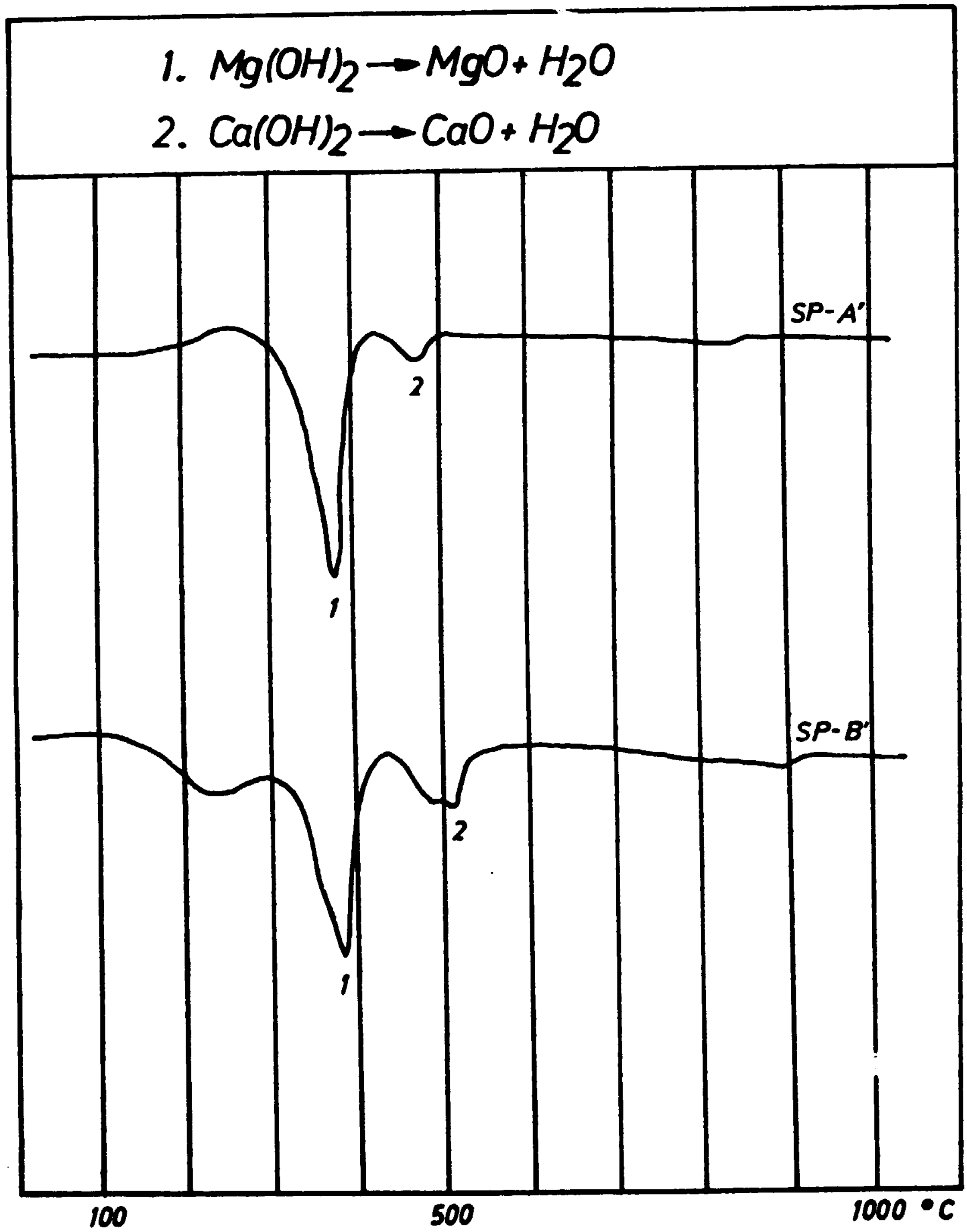

Figure 3. Thermogram of semi-products SP-A´ and SP-B´

C. Discussion on the results obtained

By determining chemical composition of semi-products SP-A (2.36-16 mm) and SP-B (1.18-2.36 mm) good agreement of results was obtained with those of investigations done in Zimbabwean institutes. Only on composite sample SP-B 4.56% of CaO was obtained instead of 5.15%, which was the mean value of investigation on several small samples. The difference occured due to sampling and partially due to investigation methods applied.

Additional screening of semi-products SP-A (2.36-16 mm) on 3 mm sieve proved useful. Semi-product SP-A´ was obtained (3-16 mm) with less than 3% of CaO. This level of CaO is to enable production of high grade dead burned magnesite, suitable for production of quality magnesite and magnesite-chrome bricks. Calcined upgraded magnesite, grade SP-B´ (1.18-3 mm), is to enable production of dead burned magnesite of normal quality, i.e. for production of magensite bricks.

By beneficiation of magnesite from Kadoma Mine by selective hydration and by screening on 1.18 and 3 mm screens recoveries and CaO contents are obtained, such as given in Table 8.

Table 8.
Efficiency and CaO content in product of magnesite beneficiation

Products	Class (mm)	Efficiency (%)	CaO content (%)
SP-A´	-16+3	35-40	2.5-3
SP-B´	-3+1.18	30-35	4-5.5
Reject	-1.18	28-32	10-15
Input:	-25+0	100	6.75

The results shown on Table 8 come from the results given in the Report: Investigations on the possibility of beneficating of local raw magnesite for the production of basic refractories, tables 21 and 22, and are based on these additional investigations of the semi-products A and B, given in tables 2, 4 and 6.

Starting from input calcined magnesite, efficiency through semi-products A nad B is about 70%, which is considered high for breccia-type magnesites.

IV. PRODUCTION OF DEAD BURNED MAGENSITE BY DIRECT DEAD BURNING

A. Production

The semi-product SP-A has a favourable grain size and lower CaO content so that tests of direct dead burning were done under stationary conditions. For dead burning a laboratory bell kiln was used and fired with a mixture of propane-butane and could reach 2200°C. On temperatures up to 1600°C air is used for combustion, and over 1600°C oxygen is used.

Samples were put into magnesite dishes and fired at 1800°C during two hours.

B. Determination of AD dead burned magnesite characteristics

After firing and cooling, determination of characteristics was done, i.e. macrospopic appearance, grain size distribution (Table 9), chemical composition (Table 10), mineralogical composition and physical properties (Table 11).

Macroscopis appearance

The obtained AD dead burned magnesite is of appearance typical for dead burned magnesites obtained of natural magnesites. It consists of light and dark gray grains of irregular shape. About 60% of grains are of light color. External appearance of grain, as well as fracure, indicate that material was well dead burned.

Table 9.
Grain size distribution
of dead burned magnesite (DBM-AD)

-16 +10 mm	6.06 %
-10 + 6 mm	23.47 %
- 6 + 4 mm	32.07 %
-4 + 2 mm	36.49 %
- 2 mm	1.91 %

Table 10.

Chemical composition of dead burned magnesite, AD, %

	Ign. loss	SiO_2 %	Al_2O_3 %	Fe_2O_3 %	CaO %	MgO %	Free CaO %
Light colour grains	0.32	0.65	0.11	0.10	2.36	96.46	0.51
Dark gray grains	0.54	0.70	0.21	0.20	2.98	95.37	0.79
AD, Total:	0.32	0.70	0.16	0.19	2.76	95.87	0.74

Mineralogical investigations

Mineralogical investigations of dead burned magnesite within this work were done in polarizytion microscope and by X-Ray diffraction method.

For microscopic investigations reflected light preparations were made. Polishing of preparate surfaces was done on STRUERS AP-2 device (Denmark), and as a polishing means suspensions of Al_2O_3 were used. Investigations were done in polarization microscope PHOTOMIKROSKOP-III, produced by OPTON (ZEISS), West Germany. Micro photographing of characteristic spots on preparates was done by a camera in microscope body, with a Black-and-White Negative film ORWO-NP22 (East Germany).

X-Ray examinations were done with X-Ray diffraction apparatus PHILJPS PW 1009/3.Investigations were done with X-Ray tube with Cu and Co anode, with 40 kV and 20 mA. For copper tube Ni filter was used, and for cobalt tube Fe filter was used. Diffractometric method was applied and reflections were registered on diffractometer PW 1051. Rate of goniometer movement was $2Q = 1^{\circ}$ a minute, and the rate of writer at difractometer was 400 mm/h.

On reflected light preparations of dead burned magnesite AD, rather big unhomogenity was observed both in periclase crystal size and sort, quantity and distribution of silicates.

With most dead burned magnesite grains, dimensions of periclase crystals range from 80 to 100 μm (figure 4). However, in grains with increased content of silicates periclase crystals dimensions are about 50 um (figure 5). In dead burned magnesite grains contain small quantity of slicates, bond among periclase grains is achieved mainly by direct contacts (see figure 4). In cases when in grains or in some their portions greater quantities of silicates are present, periclase crystals are bonded by silicates (figures 5, 6 and 7). The presence of different silicates was observed: Forsterite ($2MgO \cdot SiO_2$), Monticellite ($CaO \cdot MgO \cdot SiO_2$), Mervinite ($3CaO \cdot MgO \cdot 2SiO_2$) and dicalcijum silicate ($2CaO \cdot SiO_2$). Of these silicates, dicalcium silicate is most frequently present (figures 5 and 7). Majority of these silicates was identified by X-Ray Diffraction as well (figure 8). In some magnesite grains free CaO is present which is involved in hydration process to great extent, and Portlantide mineral was formed ($Ca(OH)2$). Calcium silicates and free CaO are present in dark gray dead burned magnesite grains. It was not possible to identify iron minerals in these grains due to a small quantity, but it could be assumed that in such mineral association they are present in a form of dicalcium ferrite.

Pores are present in comparatively small quantity and they are predominantly of isometric shapes. Irregular cracks are observed some of which are filled with smaller or greater quantity of silicates.

Figure 4.
Microphotograph of AD dead burned magnesite
Reflected light
Enlarged 100 x

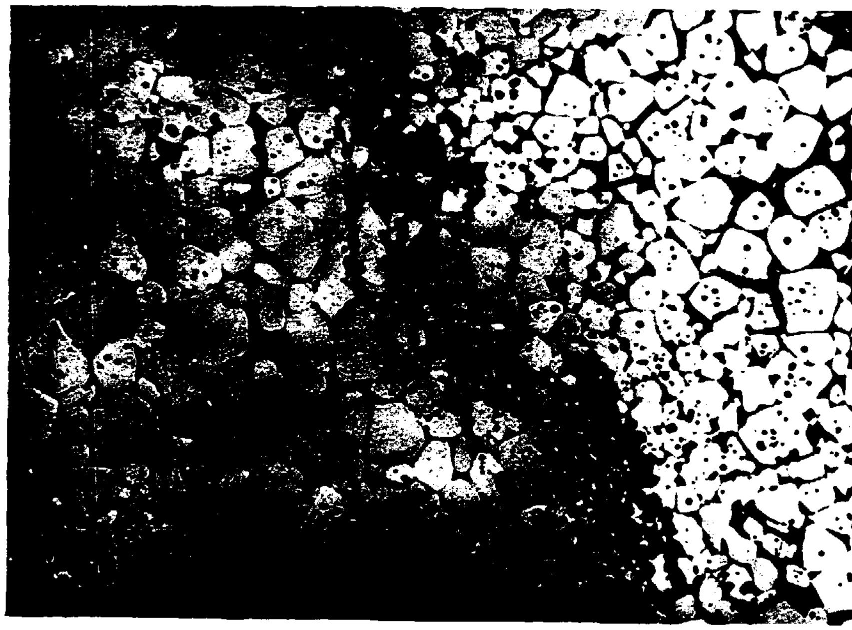

Figure 5. Microphotograph of AD d.b.meganesite
Dark magnesite grain was taken. Dicalcium silicate is between periclase crystals; clustered dark crystals in the middle are free calcium oxide; reflected light preparation; enlargement 100 x

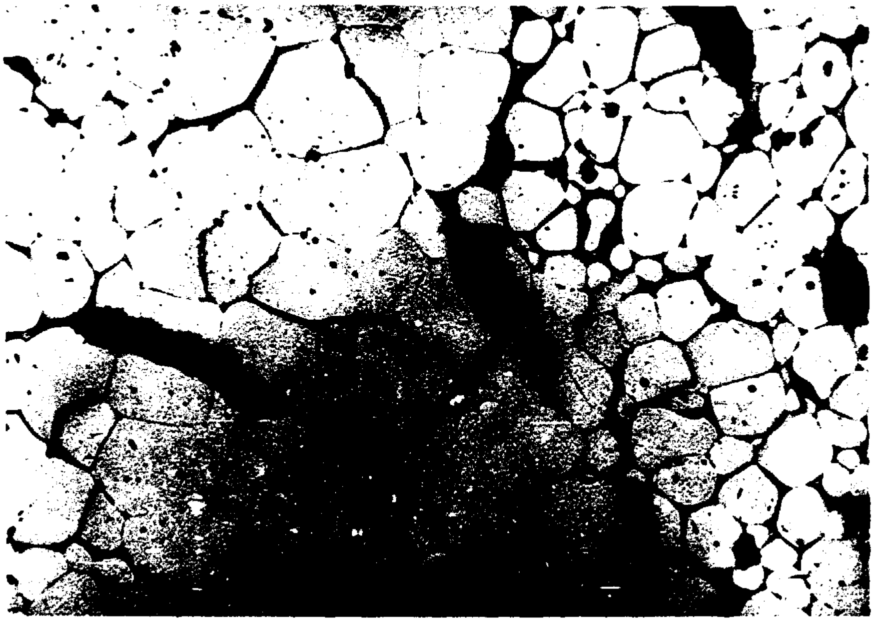

Figure 6. Microphotograph of light AD d.b. magnesite grain; magnesite grain with cilicate mainly in form of Mervinite was taken; reflected light preparation: enlargement 100 x

Figure 7. Microphotograph of light AD d.b. magensite grains;magnesite grain with dicalcium silicate was taken; reflected light preparation; enlargement 100 x

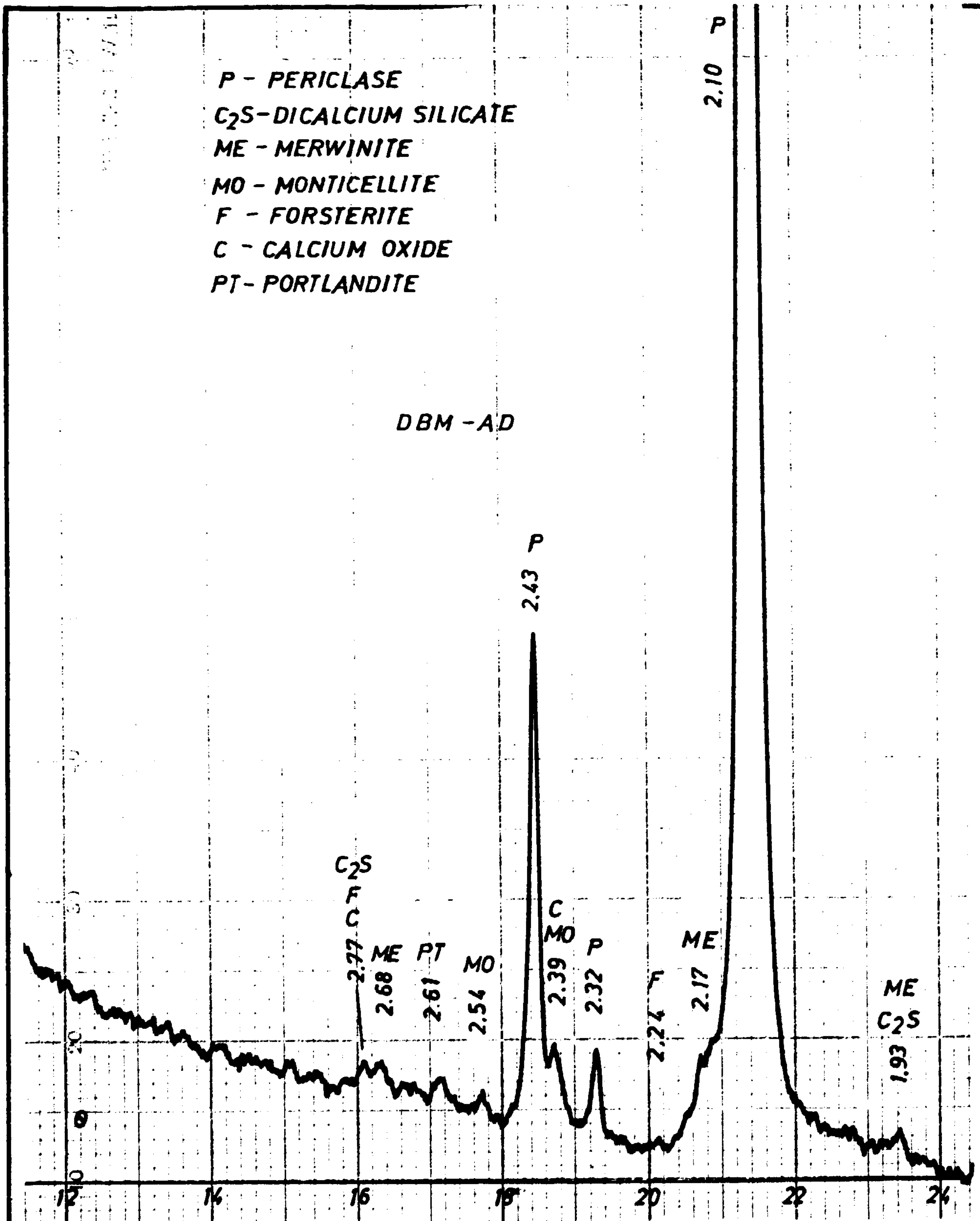

Figure 8.

X-Ray pattern of AD dead burned magnesite

Table 11.

Physical properties of AD dead burned magnesite

Density	3.56 g/cm^3
Apparent density	3.31 g/cm^3
Porosity	7.58 %
Hydration . . . . (autoclave, 0.6 bar, 4 h)	4.09 %

Density was determined according to JUS B. D8.311, and hydration was determined according to an internal Magnohrom methode, based on ASTM C544-68.

In order to determine macroscopic changes, the sample was subjected to vapour influence during 16 hours on atmospheric pressure. Appearance of AD dead burned magnesite after this testing was presented on figure 9. It was observed that at certain number of gray grains desintegration occured, while white grains remained unchanged.

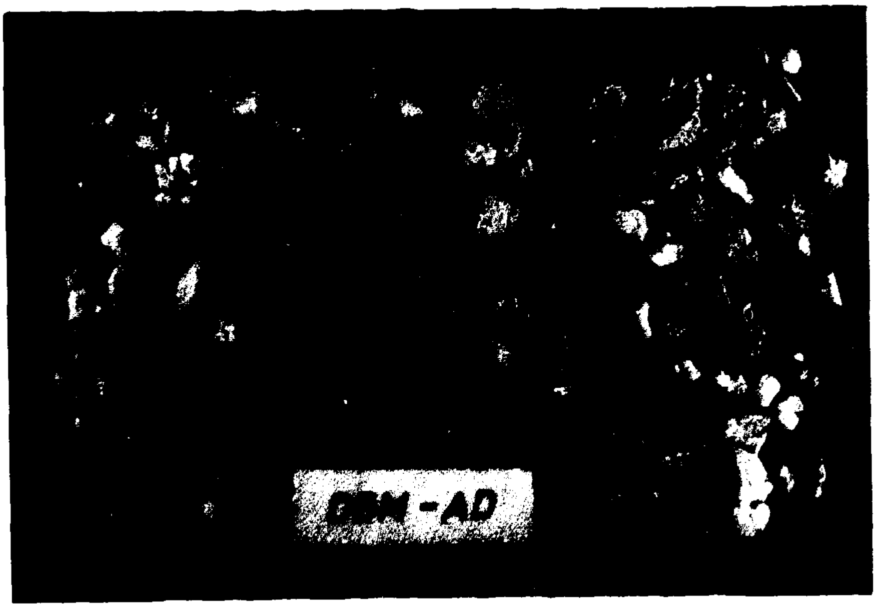

Figure 9. Appearance of AD dead burned magnesite after hydration test

C. Duscussion of results

By direct dead burning of semi-product SP-A dead burned magnesite AD was obtained, consisting of light and dark-gray irregular shaped grains.

By its chemical composition, with over 95% of MgO it ranges into high grade natural dead burned magnesites. Its disadvantages are rather heterogeneous composition, causing presence of 0.74% of free CaO. Because of hydration tendency, this free CaO can cause formation of cracks during production - on brick drying. Hydration tendency is evident only in some dark-gray grains, while light-gray grains are more hydration-resistant.

Microstructural investigations show that on firing at 1800°C good degree of dead burning was obtained. In majoriry of d.b. magnesite grains periclase crystals size is 80-100 um. Only some grains, having increased content of silicates, have periclase crystals size of about 50 um. In lighter coloured grains, in addition to periclase, forsterite, monticellite, mervinite and dicalcium silicate are also present. In dark d.b. magnesite grains, beside periclase, dicalcium silicate and free CaO are mainly present.

This d.b. magnesite has a density which is tipical for this sort of material, and good apparent density is also achieved (3.31 g/cm^3).

By direct dead burning of semi-product SP-A, dead burned magnesite AD of good quality was obtained. Its only disadvantage is heterogeneous composition, as well as high CaO/SiO_2 ratio, 3.9, containing therefore about 0.7% of free CaO.

V. PRODUCTION OF DEAD BURNED MAGNESITE BY MILLING-BRIQUETTING-DEAD BURNING PROCEDURE

By direct dead burning of semi-product SP-A´, dead burned magnesite was produced (DBM-AD) with high MgO content, but of rather heterogeneous composition and there are white and dark gray grains. The white grains, in addition to periclase (MgO), contain certain quantity of silicates, such as: Monticellite ($CaO \cdot MgO \cdot SiO_2$), Mervinite ($3CaO \cdot MgO \cdot 2SiO_2$) and dicalcium silicate ($2CaO \cdot SiO_2$).

In dark gray grains, in addition to periclase, dicalcium silicate and free CaO are mainly present. Because of such heterogeneous composition, such technological procedure should be applied which enables obtaining of dead burned magnesite of more even chemical and mineralogical composition.

The most efficient technological procedure for obtaining uniform chemical and mineralogical composition of dead burned magnesite is the procedure comprising milling, briquetting and dead burning at high temperatures. By this procedure , all phases are enabled to get in direct contact among themselves, and to react among themselves during dead burning, resulting in stable, highly refratory phases. With semi-product SP-A´, this procedure is to enable production of dead burned magnesite (DBM-A) which would primarily contsist of highly refractory periclase (MgO) minerals and of dicalcium silicates. The melting points of these minerals are 2800°C and 2130°C, respectively.

The semi-product SP-B´ is of a smaller size and with considerably higher content of CaO, and a stable dead burned magnesite (DBM-B) can be produced of it only by a procedure of fine milling with addition of SiO_2 and Fe_2O_3 ,then briquetting and dead burning. By such technological procedure regulation of mineralogical composition is enabled in semi-product SP-B´, adding other oxydes which will, first of all, bind the free CaO.

For binding extra CaO in dead burned magnesite DBM-B, iron oxide and SiO_2 were used, and the resulting d.b. magnesites bear the following designations: DBM-BF and DBM-BS.

Characteristics of dead burned magnesite obtained by this procedure are, in addition to chemical and mineralogical composition, greatly influenced by particle size, pressure of briquettes pressing and dead burning temperature. To each of these factors, due attention was paid within technological procedure of production of d.b. magnesites DBM-A, DBM-F, DBM-BF and DBM-BS.

A. Fine milling of semi-products SP-A´ and SP-B´

Particle size, particle shape and distribution are important factors on briquetting and dead burning of briquettes made of fine milled material. These values influence the main characteristics of d.b. magnesite, such as: apparent density, size and distribution of pores and periclase crystals growth. Particle size is reduced by milling; specific surface is increased at the same time and, by that, surfice energy also, which is an additional driving power for dead burning process.

For obtaining d.b. magnesites DBM-A, DBM-BF and DBM-BS, semi-products SP-A´ and SP-B´ were milled in a laboratory mill with steel balls, having volume of 36 liters, in a closed system, with no continuous discharge of already finished (milled) portion. The aim of milling was to obtain powder under 100 μm. Semi-product SP-A´ was milled without additions, and to the semi-product SP-B´, during milling, iron oxide was added in one case, and fine quartz sand in another. The obtained grain size distribution, after milling, is presented in table 12.

Table 12

Grain size distribution of the samples SP-A´, SP-B´F and SP-B´S after milling

Class (mm)	SP-A´	SP-B´F	SP-B´S	
+ 0.1	6.71	4.82	5.57	(%)
-0.1 +0.063	10.55	10.76	7.82	(%)
-0.063	82.73	84.42	86.47	(%)

The SP-B´F is the semi-product of the SP-B´ with the addition of 2.5% of iron oxide.

The SP-B´S is the semi-product of the SP-B´ with the addition of 1.5% of SiO_2.

On these powders, beside grain size distribution, packing mass and specific surface were also determined. These values are given on table 13.

Table 13

Paking mass and specific surface of the samples SP-A´, SP-B´F and SP-B´S

	SP-A´	SP-B´F	SP-B´S
Specific surface (m^2/g)	8.38	9.24	8.92
Packing mass (g/cm^3)	1.061	1.121	1.161

To check the effect of particle size of milled calcined magnesite on d.b. magnesite characteristics, some quantity of the semi-product SP-A´ was milled in vybro mill to under 40 µm. This sample (*SP-A´) was separately briquetted, dead burned and characteristics of d.b. magnesite obtained were examined.

B. Briquetting

Behaviour of material on dead burning and characteristics of d.b. magnesite are also influenced by pressure of pressing during briquetting. With increase of pressure of pressing, apparent density of green presslings (pressed bodies) also increases, shrinking on firing reduces and apparent density of fired briquetts increases.

On briquetts pressing machine briquettes were made of fine milled semi-products SP-A´, SP-B´F and SP-B´S. Quantity of material available was not sufficient to perform this operation on semi-industrial pressing machine made by Köppern, with capacity 1t/h, specific pressure up to 80 KN/cm, and small laboratory pressing machine was used, produced by Hutt, with specific pressure of 30 KN/cm. Briquetting was done without addition of binder, by multiple passing through the machine, because of low specific pressure, on first passing through of material , it was not possible to obtain compact briquettes. On passing material through pressing machine certain compacting takes place, i.e. one portion is formed into big particles and grain size distribution of starting powder is changed. By multiple passing briquettes of desired shape and hardness are obtained. This was how satisfactory percentage of whole (undamaged) briquettes (over 80%) was obtained.

A portion of semi-product SP-A´, which was milled to upper limit size of 100 μm, and a portion of semi-product*SP-A´, milled to upper limit size of 40 μm, were briquetted on laboratory briquette pressing machine with specific pressure of 50 KN/cm. This time material was passed through the machine three times. Apparent densities of "green" briquettes obtained, determined in alcohol, are given in table 14.

All of the briquetting done at room temperature.

Table 14
Apparent densities of green briquettes
SP-A´, *SP-A´, SP-B´F and SP-B´S

Sample design.	Grain size distribution (μm)	Pressure (KN/cm)	Number of passings through	Apparent density (g/cm^3)
SP-A´	100	30	6	2.37
SP-A´	100	50	2	2.41
*SP-A´	40	50	3	2.44
SP-B´F	100	30	6	2.36
SP-B´S	100	30	6	2.37

The obtained values for apparent density of green briquettes show the influence of upper limit size of powder and influence of specific pressing pressure.

C. Samples dead burning
DBM-A, DBM-BF and DBM-BS

Dead burning is a process of densification of porous body on high temperatures, in which porosity of pressed body is reduced together with dimensions reduction and mechanical hardness increasement.

A mixture of iron oxide was added to semi-product SP-B´ with the aim to bind free CaO into a stable compound. Besides, in oxydizing atmosphere, trivalent ion of Fe promotes dead burning and recrystallization of periclase. The added SiO_2 is to bind excess CaO and to take it over into a stable, highly refractory dicalcium silicate and by that to contribute higher density and resistance to hydration of briquettes, comparing to the briquettes containing free CaO.

To obtain dead burned magnesite of satisfactory apparent density, porosity and crystallization, the producead briquettes of such chemical composition have to be dead burned on the temperature over 1700°C. Dead burning of briquettes was done in laboratory bell kiln, under stationary conditions, on temperature up to 1900°C.

Briquettes were put into magnesite dishes and introduced into cold kiln. DBM-A and DBM-F briquettes are test dead burned on 1700, 1800 and 1900°C. As the obtained characteristics of DBM-A and DBM-BF at 1800°C are satisfactory, and only slightly better at 1900°C, it was decided to treat the rest of the briquettes and all DMB-BS ones, at 1800°C. Holding time at maximum temperature was 2 hours.

D. <u>Determination d.b. magnesite characteristics DBM-A, DBM-BF and DBM-BS</u>

After stationary dead burning and cooling, determination of characteristics of d.b. magnesite obtained (DBM-A, DBM-BF and DBM-BS) was done, i.e. chemical composition (Table 15), mineralogical investigations and physical properties (Table 16).

Macroscopic view of briquettes DBM-A, DBM-BF and DBM-BS, after dead burning at 1800°C is presented at figure 10.

Figure 10. Macroscopic view of d.b. magnesites DBM-A, DBM-BF and DBM-BS after dead burning at 1800°C

Table 15.

Chemical composition of d.b. magnesites DBM-A, DBM-BF and DBM-BS (in %)

Design.	Loss on ignition	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	B_2O_3	Free CaO
DBM-A	0.23	0.68	0.12	0.20	2.62	96.13	0.02	0.15
DBM-BF	0.34	0.83	0.14	2.80	4.94	90.93	0.02	0.19
DBM-BS	0.22	2.17	0.22	0.19	4.81	92.37	0.02	0.10

Mineralogical investigations of dead burned briquettes: DBM-A*, DBM-BF and DBM-BS

DBM-A Briquettes

Mineralogical investigations of these briquettes, as well as the other ones within this work, were done in polarysation microscope and by X-Ray diffraction method.

The DBM-A briquettes were fired on 1700, 1800 and 1900°C. Dimensions of periclase crystals in briquettes that were fired at 1700 °C are about 55 µm (figure 11). With increase of firing temperature, crystals dimensions also increase, so that after firing at 1800°C thea are about 70 µm (figure 12). After firing at 1900°C crystal dimensions were not changed much; the measured increase was about 5-10 µm in relation to firing at 1800°C (figure 13).

By increasing temperature of briquette firing, number of pores is reduced or pores are enlarged by joining. After firing at 1900°C majority of pores have dimensions similar to dimensions of some periclase crystals (figure 13). On basis of these investigations it could be stated that, disregarding firing temperature, total quantity of pores is not big and it does not change much with increasment of firing temperature.

In all briquettes, regardless the temperatures of firing, calcium silicates are present - dicalcium silicate and tricalcium silicate. The silicates are present in small qunatities and are rather evenly distributed between periclase crystals. Their presence was determined by X-Ray investigations (figure 14). On separate spots, very rearly, after formation of silicates, very small quantity of retained free CaO was discovered.

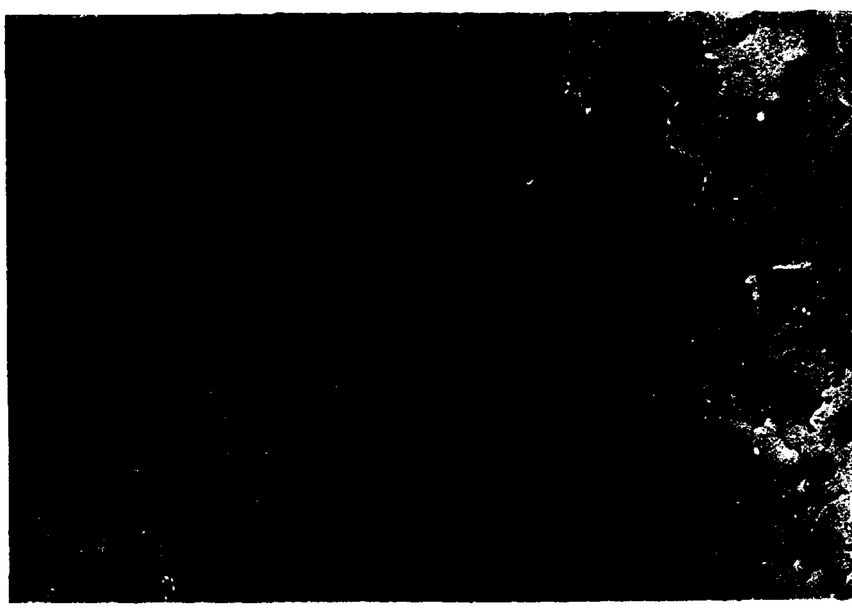

Figure 11. Microphotograph of DBM-A briquette after firing at 1700°C; reflected light; enlargement 100 x

Figure 12. Microphotograph of DBM-A briquette after firing at 1800°C; reflected light; enlargement 100 x

Figure 13. Microphotograph of DBM-A briquette after firing at 1900°C; reflected light; enlargement 100 x

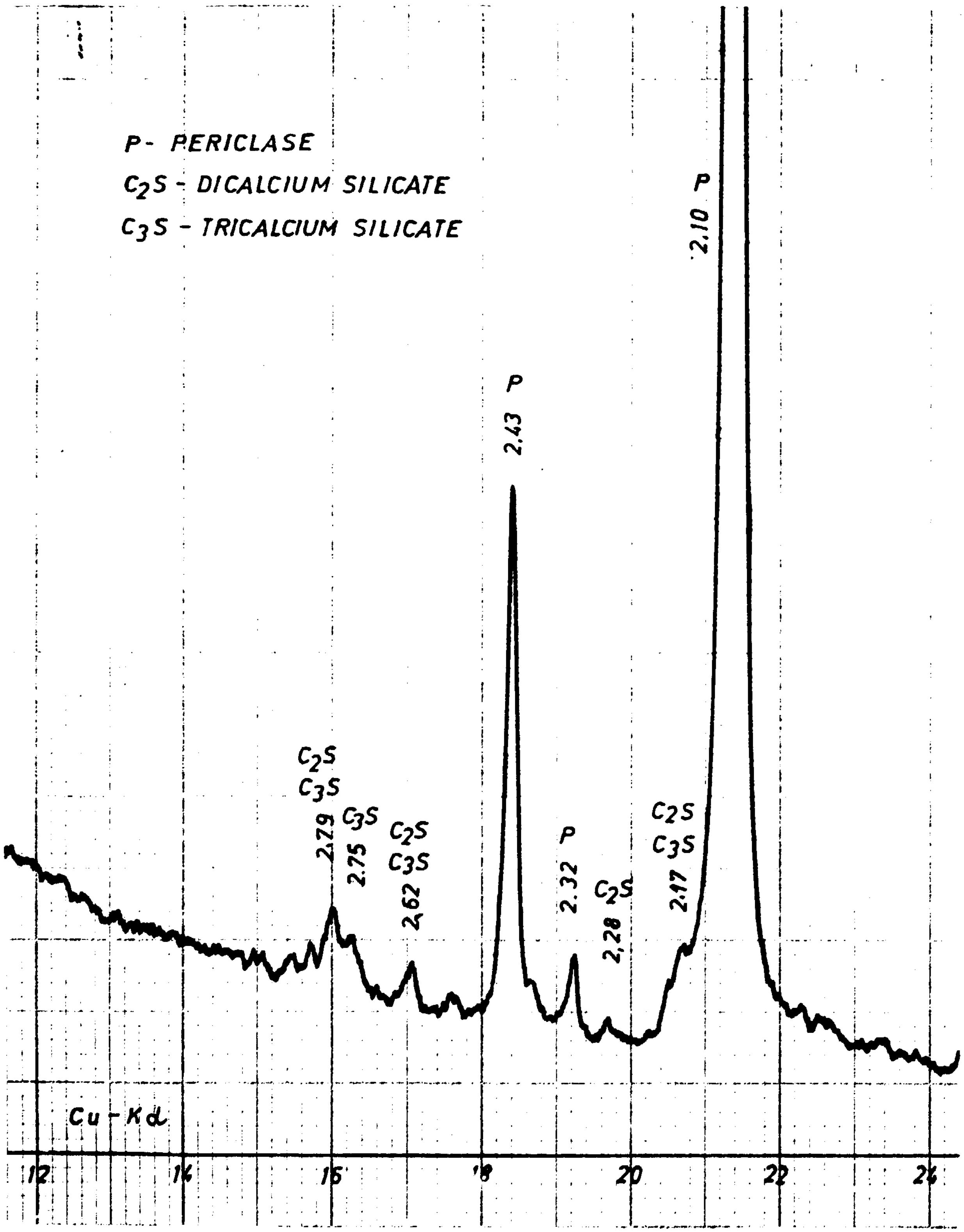

Figure 14. X-Ray pattern of DBM-A briquette after firing at 1800°C

DBM-A* Beiquettes

Briquettes of this designation were produced of the same material as DBM-A briquettes. DBM-A* briquettes were obtained by pressing of fine powdered materijal (< 40 μm) under the pressure of 50 KN/cm and were fired at the temperature of 1800°C. Dimensions of periclase crystals are about 75 μm (figure 15).

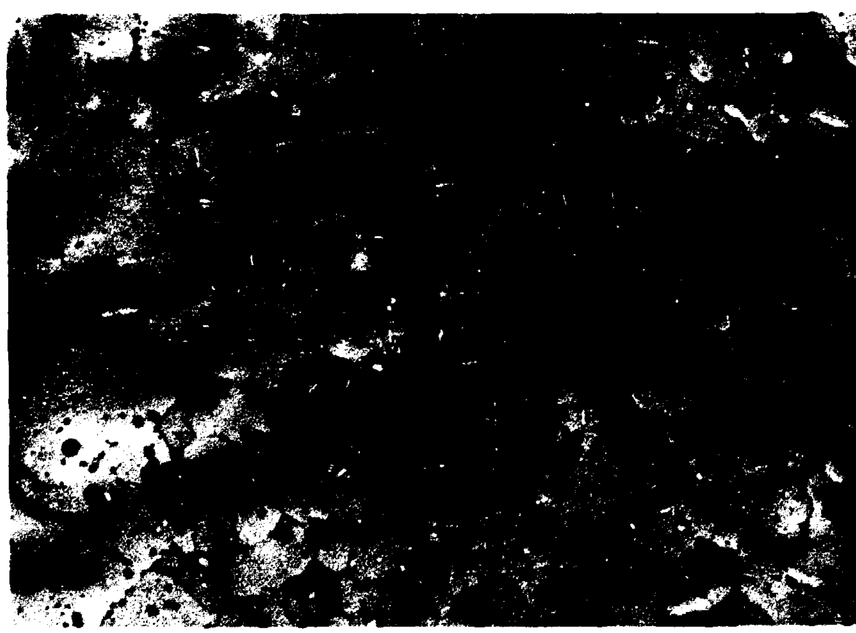

Figure 15. Microphotograph of DBM-A* briquettes after firing at 1800°C; reflected light; enlargement 100 x

By inspection of large number of microscopic prepatares of this briquette it was stated that quantity of pores is somewhat smaller compared to DBM-A briquettes. In DBM-A* briquettes comparatively smaller quantity of silicates is present which are therefore difficult to recognize by microscope.

Dicalcium silicate and tricalciumsilicate are present. These silicates are among periclase crystals, most frequently in spaces where three periclase crystals meet.

In briquettes DBM-A and DBM-A* bond between periclase crystals is predominantly direct, periclase-periclase.

DBM-BF Briquettes

These briquettes were fired at 1700, 1800 and 1900°C. After firing at 1700°C dimensions of periclase crystals were about 60 µm (figure 16). With the increase of temperature of firing considerable increase of periclase crystals dimensions occured, so that after firing at 1800°C dimensions of these crystals already were about 100 µm (figure 17). After firing at 1900°C periclase crystals dimensions are about 110 µm (figure 18).

On basis of these results it could be said that addition of iron oxide into briquettes greatly promoted the process of dead burning, especially at the temperatures of 1800°C and 1900°C. During dead burning, with increase of temperature enlargement of pores by joining occured in briquettes, but we can not say that dead burning processes at high temperatures caused shrinkage of total volume of pores (figures 16, 17 and 18).

In microscopic preparation and by X-Ray investigations as well (figure 19), the presence of periclase, tricalcijum silicate, dicalcium silicate and dicalcium ferrite was discovered. By these methods the presence of free CaO was not observed. Silicates and dicalcium ferrite fill interspaces of periclase crystals binding them in that way. However, at meny places direct bond periclase-periclase was observed.

On basis of this mineralogical composition it can be stated that addition of iron oxide in reaction with free CaO formed dicalcium ferrite and that, because of this reaction instead of tricalcium silicate only, silicate association was formed of tricalcium and dicalcium silicates in briquettes. However, the most important part of these reactions is that iron oxide, binding itself to the free CaO, enabled that, after firing, only negligible content of free CaO remained in briquettes.

Figure 16. Microphotograph of DBM-BF briquettes after firing at 1700°C; reflected light; enlargement 100 x

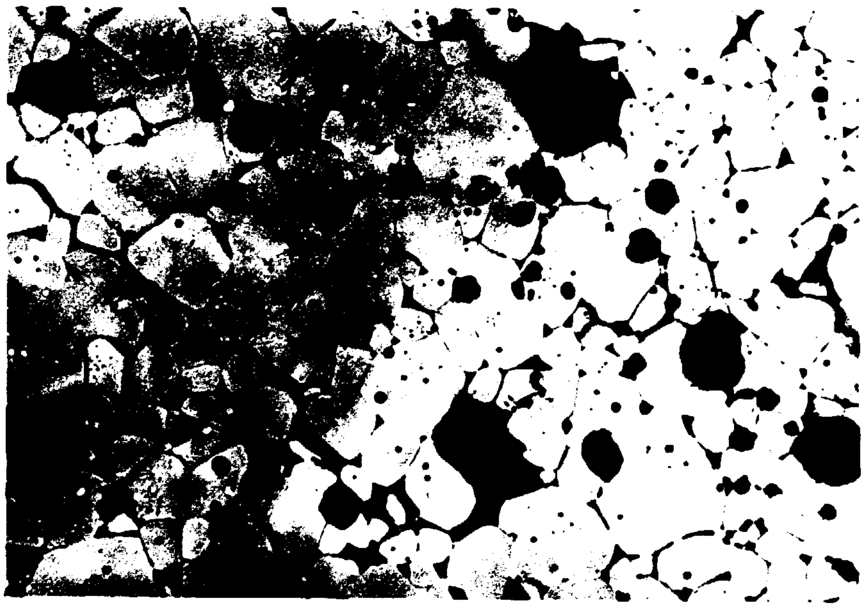

Figure 17. Microphotograph of DBM-BF briquettes after firing at 1800°C; reflected loght; enlargement 100 x

Figure 18. Microphotogra... DBM-BF briquettes after firing at 1900°C; reflected light; enlargement 100 x

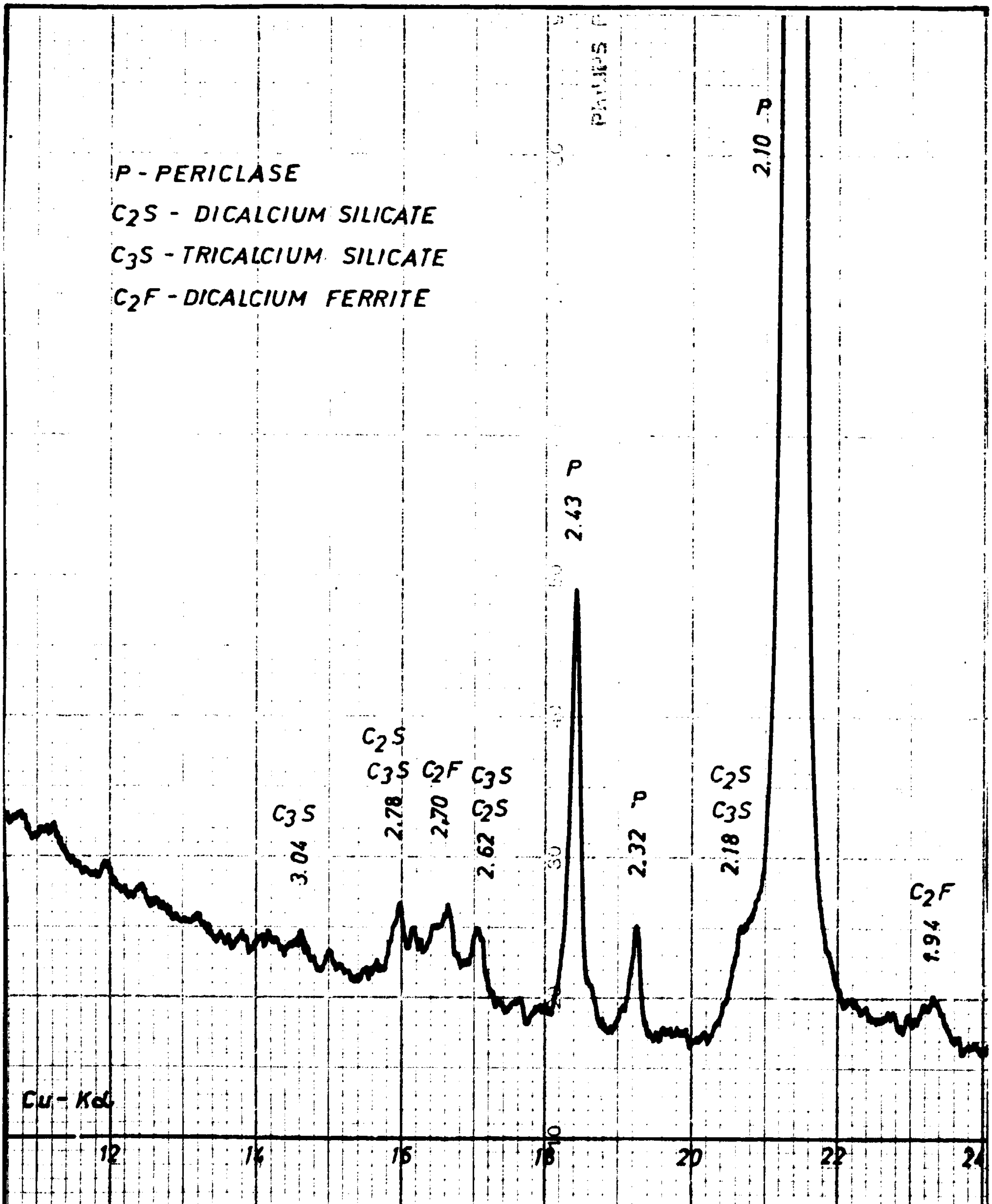

Figure 19. X-Ray pattern of DBM-BF briquettes after firing at 1800°C

DBM-BS Briquettes

These briquettes were obtained by firing green briquettes at 1800 °C. Into this mixture 1.5% of finely powdered quartz (SiO_2) was added in order to bind free CaO.

In microscopic preparations it was found that periclase crystals dimensions are about 70 μm (figure 20). Volume of pores, their shapes, dimensions and distribution are similar to those of DBM--BF briquettes, Dimensions of coarser pores are similar do those of periclase crystals.

By microscopic examination (figure 20) and by method of X-Ray difraction (figure 21) presence of prericlase and dicalcium silicates in briquettes was observed. By these methods presence of free CaO was not observed. Dicalcium silicate is found among periclase crystals having a roll of highly refractory ceramic bond. However, bond among numerous periclase crystals is direct, i.e. it is achieved by direct contact periclase-periclase.

By such mineralogical composition it can be concluded that at firing temperatures intensive reaction occured between the added quartz and the free CaO. Addition of quartz reduced CaO/SiO_2 ratio in briquettes, and dicalcium silicate was formed in the mentioned reaction.

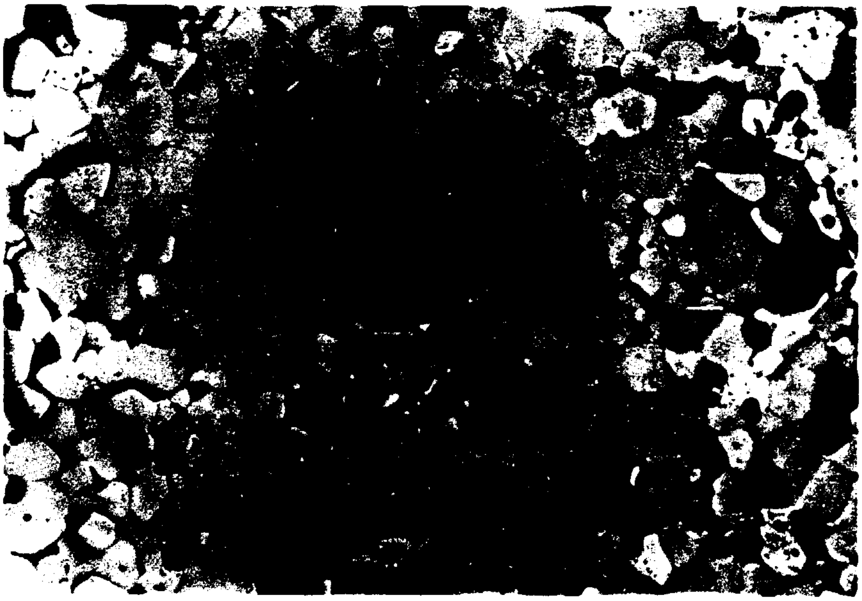

Figure 20. Microphotograph of dead burned magnesite DBM-BS after firing at 1800°C; reflected light; enlargement 100 x

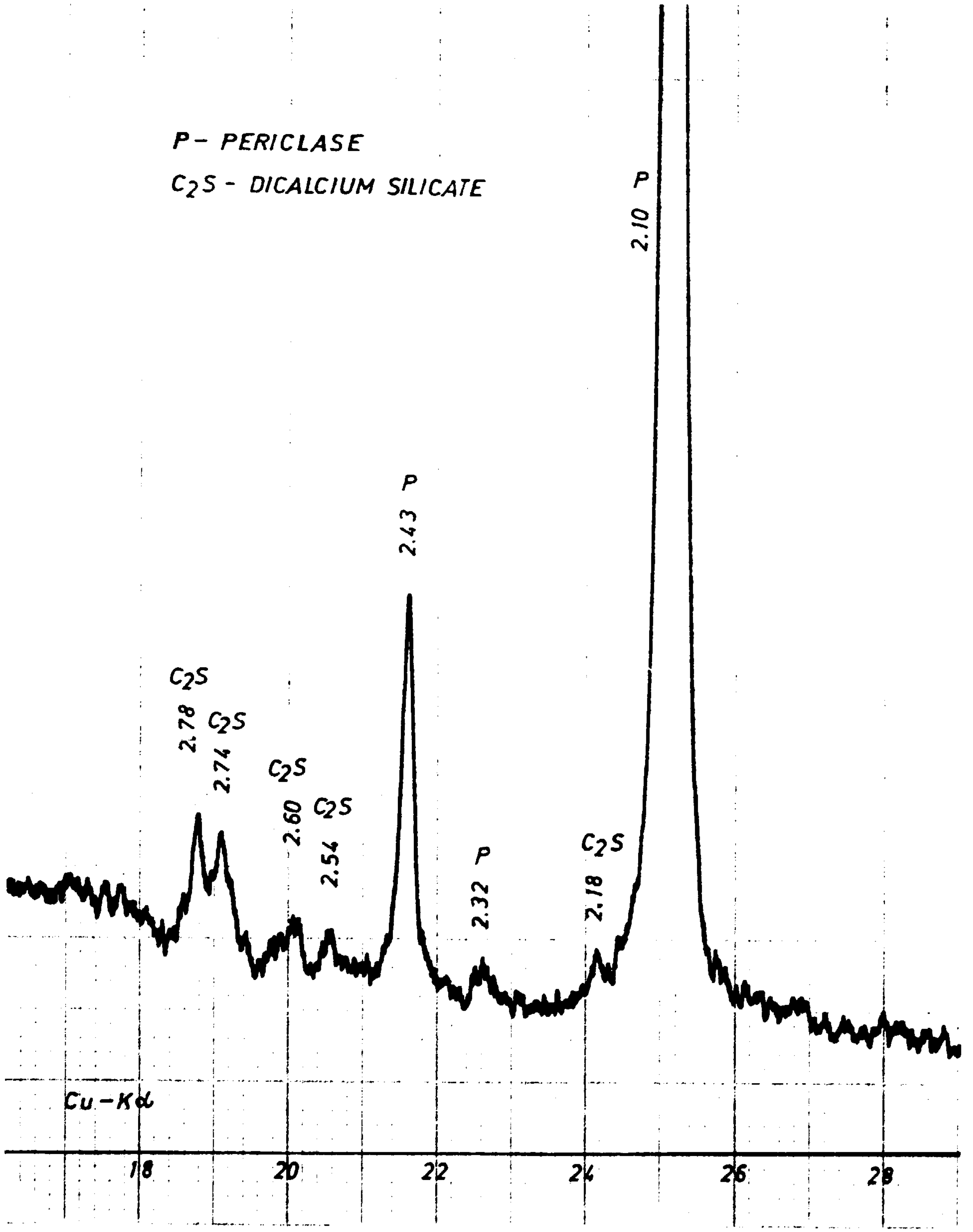

Figure 21. X-Ray pattern of DBM-BS briquettes after firing at 1800°C

Table 16

Physical properties of d.b. magnesite DBM-A burned at 1700, 1800 and 1900°C and DBM-BF and DBM-BS burned at 1800°C

	1700°C	1800°C	1800°C	1900°C	1800°C	1800°C
	DBM-A	DBM-A	DBM-A*	DBM-A	DBM-BF	DBM-BS
Density (g/cm3)	3.53	3.54	3.55	3.55	3.57	3.54
Apparent density (g/cm^3)	3.27	3.32	3.37	3.33	3.27	3.25
Porosity,%	7.36	6.21	5.07	6.20	8.40	8.19
Hydration, % (autoclav, 0.6 bar)	5.70	4.53	2.52	4.81	1.94	2.68

DBM-A* = Milling to - 40 um and pressing at 50 KN/cm

Macroscipic view of briquettes after testing of subjection to water vapour during 16 h at atmospheric pressure, is presented at figure 22.

Figure 22. Macroscopic view of d.b. magnesite DBM-A, DBM-BF and DBM-BS after hydration test

E. DISCUSSION OF RESULTS

The main aim of treatment of semi-product SP-A´ into dead burned magnesite (DBM-A) by a procedure of fine milling, briquetting and dead burning was obtaining of dead burned magnesite of homogeneous chemical and mineralogical compositions and good physical properties.

The procedure applied enabled fine particles of CaO to come into contact with other compounds, carriers of SiO_2, Fe_2O_3 and Al_2O_3, but primarily with SiO_2, and to form stable refractory compounds.
The obtained d.b. magnesite , DBM-A, is homogeneous, with high MgO content (96.13%) and low free CaO content (0.15%). Resistance of d.b. magnesite to hydration is good.

Mineralogical investigations showed that in this d.b. magnesite highly refractory minerals were present, such as: periclase (MgO), dicalcium silicate ($2CaO.SiO_2$) and tricalcium silicate ($3CaO-SiO_2$). By inspecting microstructure of briquettes fired at 1700, 1800 i 1900°C it was found that corresponding mean sizes of periclase crystals of about 55, 77 and 90 µm, respectively. It means that at 1800°C satisfactory degree of dead burning was already obtained.

D.B. magnesite (DBM-A) was obtained by briquetting at specific pressure of 30 KN/cm and burned at 1800°C. It has good apparent density of 3.32 g/cm^3. When fineness of milling was increased and specific pressure as well, to 55 KN/cm, dead burned magnesite (DBM-A* was obtained with very good apparent density - 3.37 g/cm^3.

The results obtained show that with d.b. magnesite DBM-A main aim was achieved and material of homogeneous chemical and mineralogical composition produced having good phaysical properties as well.

With MgO content of slightly over 95% and apparent density of 3.37 g/cm^3 this d.b. magnesite belongs to the high grade ones, obtained by natural magnesites processing.

As the semi-product SP-B´is of a finner grain size distribution and with higher CaO content, obtaining of more stable d.b. magnesite DBM-B is possible only by applying the procedure of fine milling,adding of suitable aditives, homogenization, briquetting and dead burning.

Because of higher CaO content and unfavourable CaO/SiO_2, Al_2O_3 and Fe_2O_3 ratios, it is necessary to add iron oxides or SiO_2 during homogenization process, in order to bind excess of free CaO and transfer it to stable refractory mineral phases. The addition of iron oxide, beside binding of free CaO, promotes the dead burning process and increases d.b. magnesite resistance to hydration, but leads to formation inferrior refractory dicalcium ferrite ($2CaO.Fe_2O_3$) as well.

By addition of SiO_2 , binding the CaO into highly refractory dicalcium silicate is enabled.

With these additions, dead burned magnesites DBM-BF and DBM-BS were produced.

DBM-BF contains 90.93% of MgO, 0.83% of SiO_2 and 2.80% of Fe_2O_3. Its hydration resistance is very good. This d.b. magnesite consists of periclase, dicalcium and tricalcium silicates and dicalcijum ferrite.

A good degree of firing was achieved by firing at 1800°C and mean crystal size was 100 μm.

By briqetting under the pressure of 30 KN/cm and by firing at 1800°C a satisfactory apparent density was achieved, 3.27 g/cm^3. By finner milling and by applying higher specific pressure on briquetting, apparent density of 3.3 g/cm^3 can be expected.

With these characteristics the d.b. magnesite ranges in mean qualities produced of natural magensites.

D.B. magnesite DBM-BS produced with the addition of SiO_2 contains 92.37% of MgO, 2.17% of SiO_2 and 0.19% of Fe_2O_3. Hydration resistance is satisfactory.

The main mineral components of this d.b. magnesite are periclase and dicalcium silicate.

By firing at 1800°C good results were obtained and mean size of periclase crystals was about 70 μm.

By briquetting under specific pressure of 30 KN/cm and by firing at 1800°C good apparent density was obtained, 3.25 g/cm^3.

By finner milling and application of specific pressure of over 50 KN/cm, by firing at 1800°C , apparent density of about 3.3 g/cm^3 is expected.

With these characteristics this d.b. magnesite ranges in mean qualities produced of natural magnesites.

VI. PRODUCTION OF TEST MAGNESITE BRICKS M-A, M-BF and M-BS

Test magnesite bricks, in grades M-A, M-BF and M-BS were made of dead burned magensites DBM-A, DBM-BF and DBM-BS. Characteristics of these bricks were investigated on bases of which evaluation of obtained brick qualities were done.

A. Production

Briquettes of d.b. magnesite DBM-A, DBM-BF and DBM-BS were obtained in the following qunatities:

DBM-A . . . . 25 kgs
DBM-BF . . . . 32 kgs
DBM-BS . . . . 17 kgs.

Test magnesite bricks were produced of each d.b. magnesite separately, with no inter-combinations of different magnesite qualities. It is to be noted that during production of test bricks, as quantity of starting components was small, it was not possible to try several alternative procedures for each brick type. According to literature and own practical experience, procedure of production had to be choosen without later corrections in respect of desired improvement of some characteristics of test bricks.

1. Preparation of components

Grain component of each d.b. magnesite was prepared separately by crushing in jaw crusher, while crushing process was so performed as to obtain inter-fraction ratio of d.b. magnesite components with grain size of 0-4 mm, to enable maximum of dense packin in a brick. Fine component of d.b. magnesite (-0.1 mm) was prepared in a laboratory ball mill.

2. Preparation of mixtures for pressing of bricks

Mixture for pressing was prepared with optimul combining of obtained classes of d.b. magnesites, with the addition of binding agent. Into a mixer, type "Eirich", granular component was added (0-4 mm). After through mixing, magnesium sulphate solution was added as a binding agent. When all grains were wetted, fine fraction was added (-0.1 mm). Then the prepared mixture was additionally mixed to make it completely homogeneous.

3. Pressing the test bricks

After having been homogenized, the prepared mixture was pressed in a standard form "2", in an oil-hydraulic pressing machine, with double pressing, with unit pressure of 100 N/mm^2, with one deaeration during the pressing operation.

4. Drying the pressed bricks

The pressed bricks were dried by gradual heating of bricks up to 120^oC and by holding them at that temperature for 24 hours. After drying, bricks of all grades had satisfactory mechanical strength and had no cracks.

5. Firing of test bricks

The bricks were fired in industrial tunnel kiln with burner system adapted for alternate usage of oil and gas for drive energy. Schedule of firing was adapted to magnesite bricks, and maximum temperature was 1720^oC.

B. Testing of characteristics

As quantity of obtained test bricks was small, it was necessary to choose characteristics that would give the best insight in quality of test magnesite bricks to be tested. Each characteristic of all brick grades was tested on at least two samples, in order to be able to check obtained values and avoid possibe mistakes in testing procedure.

1. Macroscopic view of test bricks

After firing M-A grade bricks had characteristic greenish-yellow color, smooth surfaces, good edges and no cracks. Macroscopic section indicated good composition of brick and a solid structure (figure 23).

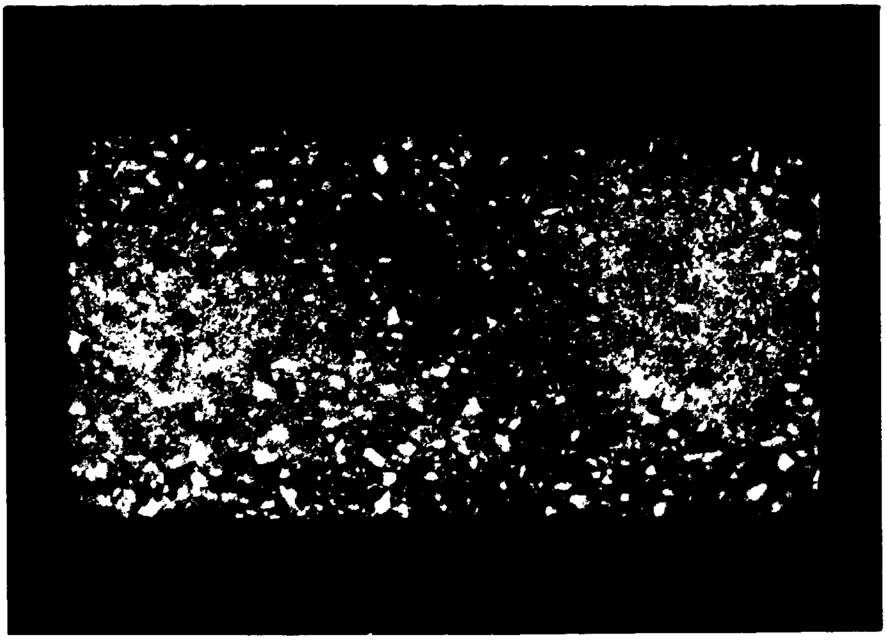

Figure 23. Macroscopic section of M-A grade of magnesite brick

M-BF grade bricks after firing had dark brown color, smooth surfaces and good edges. Macroscopic section of bricks indicated extremely compact structure of brick with low porosity and good density (figure 24).

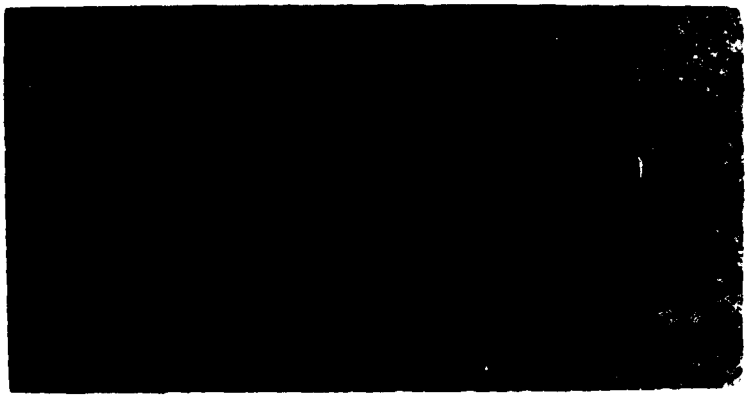

Figure 24. Macroscopic section of M-BF grade of magnesite brick

M-BS grade bricks after firing had yellow-greenish color, good edges and no cracks. Macroscopic section indicated hardness with slightly rised porosity compared to M-A and M-BF grades (figure 25).

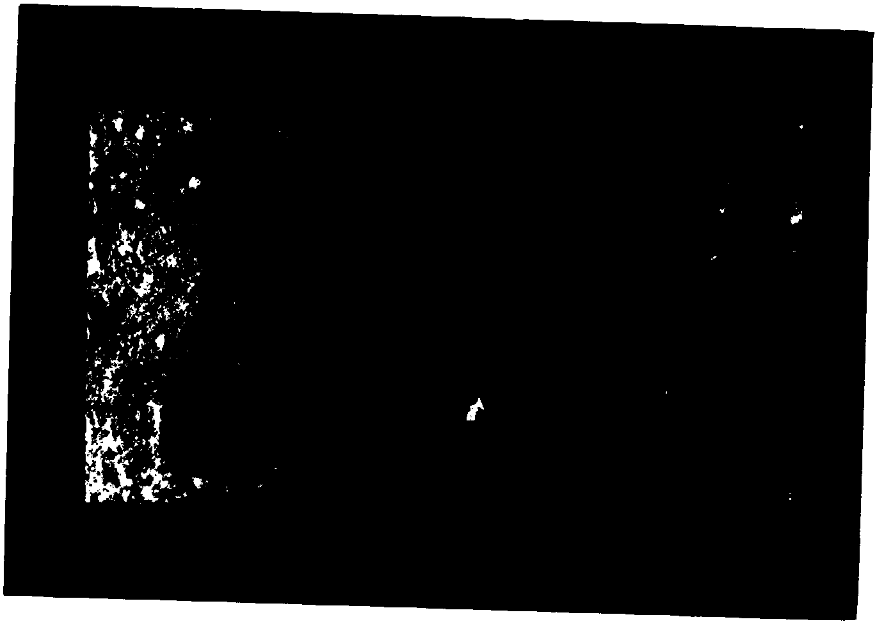

Figure 25. Macroscopic section of M-BS grade of magnesite brick

2. Chemical composition of test bricks

Chemical composition of bricks was given as mean value of several testings within the same quality. As all bricks were manufactured on basis of only one d.b. magnesite, chemical composition could not differ much from the starting d.b. magnesite.

Table 17.

Chemical composition of M-A, M-BF and M-BS grades of magnesite bricks

Grade/ oxide	M-A	M-BF	M-BS
Ignition loss, %	0.19	0.30	0.43
Fe_2O_3, %	0.31	2.28	0.41
Al_2O_3, %	0.20	0.27	0.40
SiO_2, %	0.86	0.65	2.18
CaO, %	2.82	4.40	4.74
MgO, %	95.62	92.10	91.85
B_2O_3, %	0.020	0.019	0.024

3. Physical properties of test bricks

Physical properties of bricks were given as a mean value of at least two values for one quality, on table 18.

Table 18.

Physial properties of test magensite bricks, grades M-A, M-BF and M-BS

Quality/ property	M-A	M-BF	M-BS
Apparent porosity, %	17.0	14.4	20.0
Absorption of H_2O, %	5.9	4.8	7.2
Apparent density, g/cm^3	2.89	2.96	2.83
Density, g/cm^3	3.53	3.55	3.52
Crushing strength at 20°C, N/mm^2	45.8	68.0	37.0
Crushing strength at 1500°C, N/mm^2	26.0	25.0	26.0
Refractoriness under load, Ta, °C	+1700	+1700	+1700
Thermal shock resistance, Tw	15	29	7.0
Modulus of rupture at 1400°C, N/mm^2	10.5	-	7.0
Linear thermal expansion at 20-1400°C, %			
up to 1000°C	1.33	1.73	1.37
up to 1400°C	1.79	1.77	1.75

The obtained physical properties of bricks correspond to typical values for magnesite bricks of higher and medium grades, and some of the properties are even above those values.

For determining physical properties the following methods were employed:

- For apparent porosity, adsorption of H_2O and apparent density - JUS B.D8.312;
- For brick density determination - JUS B. D8 302;
- For crushing strength at high temperatures determination - - internal method of "Magnohrom" Industry;
- For modulus of rupture determination - BS 1902 PART A;
- For refractoriness under load determination - JUS B.D8.303;
- For linear thermal expansion determination - internal method on apparatus produced by NETZSCH, with rate of heating of 5°C/min, sample ∅ 50 x 50 mm.

4. Mineralogical investigations of M-A, M-BF and M-BS bricks

The investigations were performed with the aim to identify mineral phases in bricks and to analyse their structural characteristics. Investigations were done in polarization microscope and X-Ray diffraction apparatus. For microscopic investigations reflected light preparations were made.

Magnesite brick M-A

In microscopic preparations, grain structure of brick is clearly observed, characterized by presence of coarser d.b. magnesite grains and fine-grained matrix between these grains. Boundary betwwen coarser d.b. magnesite grains and matrix is rather clear (figures 26 and 27). Dimensions of periclase crystals in coarser d.b. magnesite grains are about 70 μm, and they are somewhat smaller in matrix.

In coarse d.b. magnesite grains very small amount of silicates is present which makes their identification more difficult. Slightly bigger quantity of silicates is present in matrix, which enabled identification of dicalcium silicate and tricalcium silicate. The presence of these calcium silicates was confi... by X-Ray patterns as well (figure 28).

Slightly bigger quantity of pores is present in matrix compared to coarser d.b. magnesite garins, which is usual characteristic of this kind of bricks. Dimensions of pores in d.b. magnesite grains, measured in microscope are about 2-100 µm. Larger pores often are as big as periclase crystals. In d.b. magnesite grains majority of pores are about 20 µm. Pore dimensions in brick matrix mainly range from 5 to 300 um, and majority of them are of about 20 um. A numer of the largest pores in matrix were created by falling of material out of a brick during preparation of microscope preparation.

Bond between periclase crystals was mainly achieved by direct reaction contacts periclase-periclase. Bond between periclase crystals in a matrix, as well as between these crystals in coarser d.b. magnesite grains, is often achieved over highly refractory calcium silicates as well.

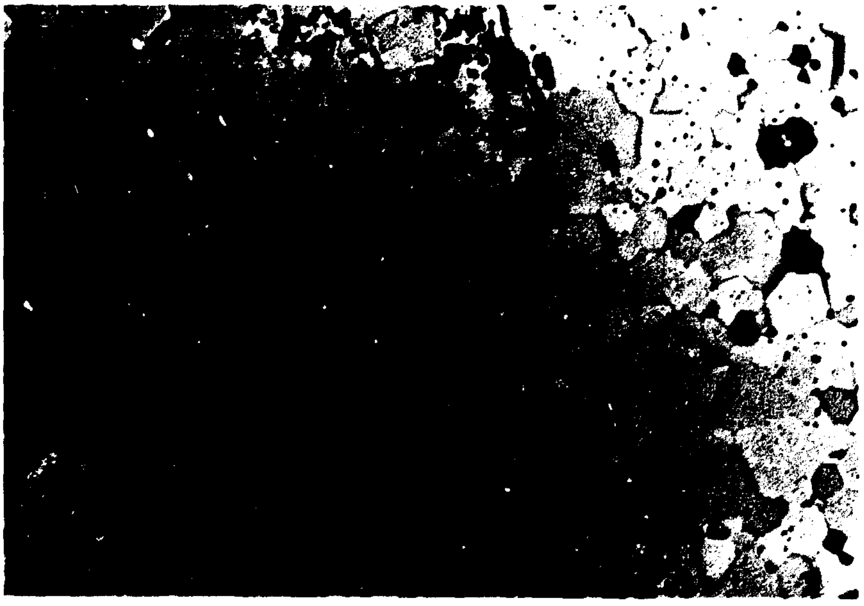

Figure 26. Microphotograph of magnesite brick M-A; Brick matrix betwwen two coarse grains of d.b. magnesite was taken; reflected light preparation; enlargement 100 x

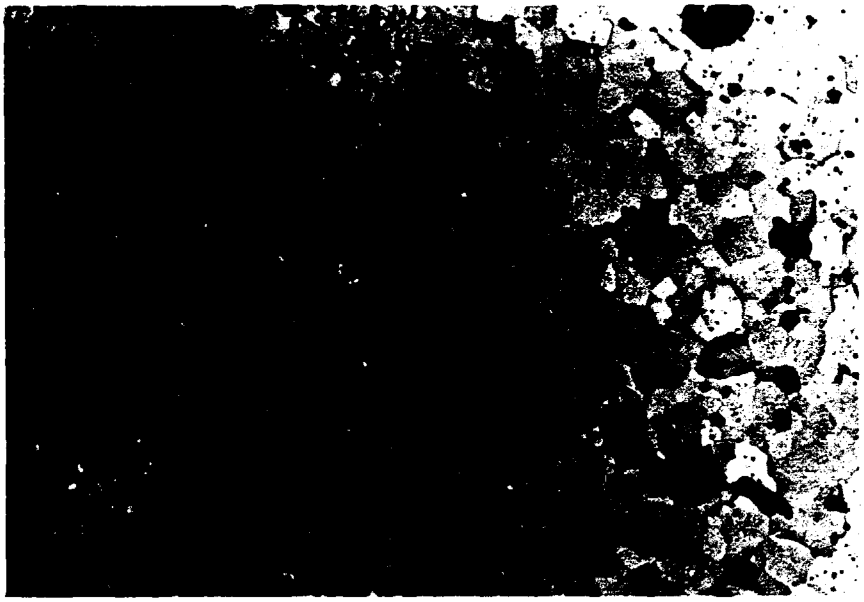

Figure 27. Microphotograph of magnesite brick M-A; brick matrix between two coarser d.b. magnesite grains was taken; reflected light preparation; enlargement 100 x

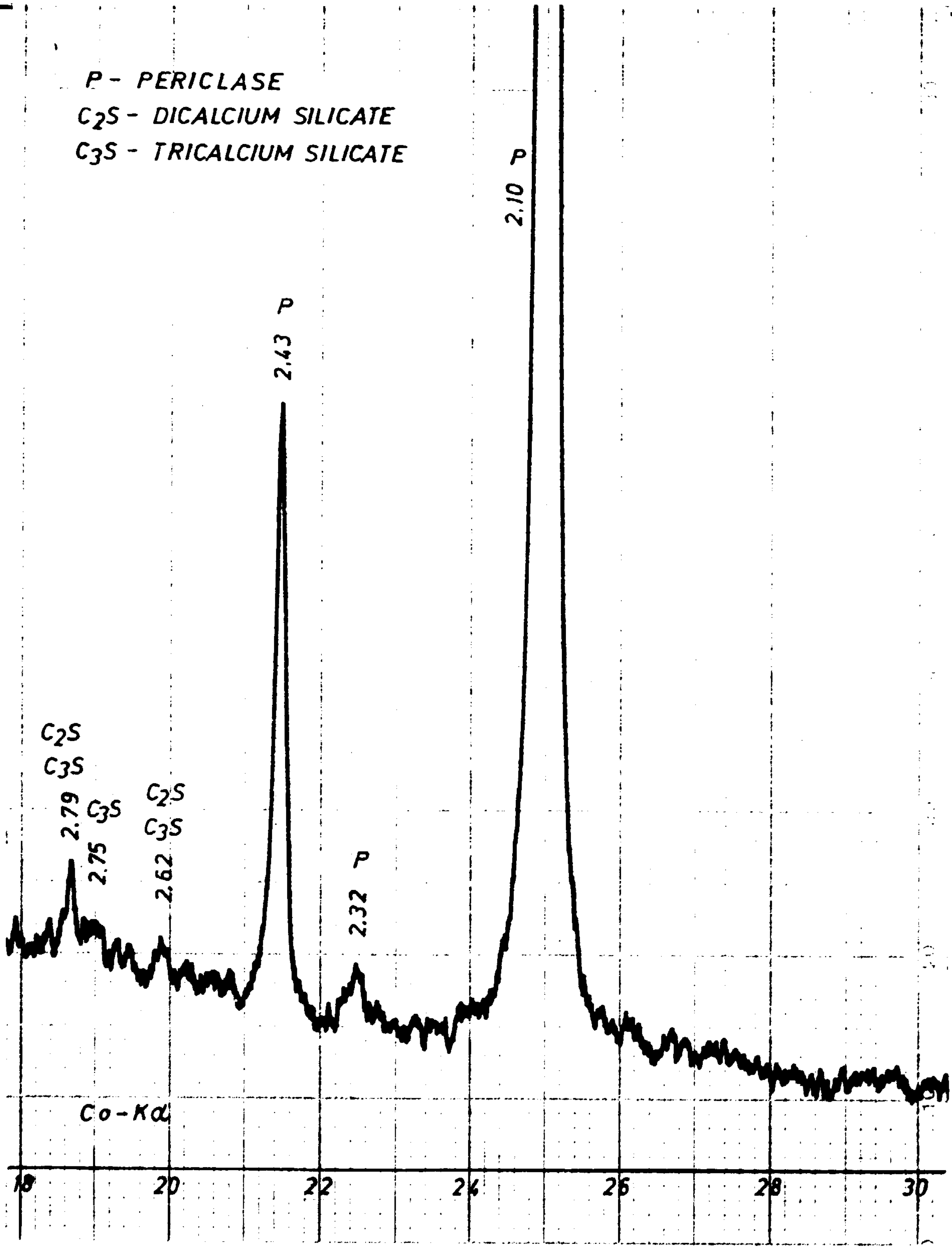

Figure 28. X-Ray pattern of M-A magnesite brick

Magnesite brick M-BF

By examination of this brick in microscopic preparations it was found that grain structure of brick was not clearly defined, because baundaries between coarse grains of d.b. magnesite and brick matrix were hard to observe (figures 29 and 30). This structure was created as a result of intensive reactions during firing. It was, primarilly, the result of periclase recrystallization which took place by influence of iron oxide present as iron-containing liquid at elevated temperatures. Dimensions of periclase crystals in coarse grains of d.b. magnesite are about 160 μm. Dimensions of periclase crystals in matrix are smaller, usually from 60 to 90 μm.

Very small amount of silicates in the form of dicalcium silicate is present in the brick. There are more of them in brick matrix than i coarse grains. Some dicalcium ferrite is present between periclase crystals. The presence of calcium silicates and dicalcium ferrites was registered by X-Ray diffraction as well (figure 31). There were no reflections on X-Ray patterns which would indicate presence of free calcium oxide.

There are more pores in matrix than in coarser grains of d.b. magnesite.

Measurements of pores in d.b. magnesite grains have shown that their dimensions range from 10 to 150 μm, and that majority is of 50 μm. Pores are somewhat larger in matrix - 20-200 μm. Majority of pores in matrix are about 70 μm. The 200 μm pores in matrix were crated during making microscopic preparation.

Bond between periclyse crystals was achieved by calcium silicates (dicalcium silicate and tricalcium silicate), dicalcium ferrites, a nd, to some extent by direct periclase-prericlase bond. Roll of low-refractory dicalcium ferrite in bonding periclase crystals is small because of big roll of highly refractory calcium silicates and because of formation of direct pericalse-preiclase bond.

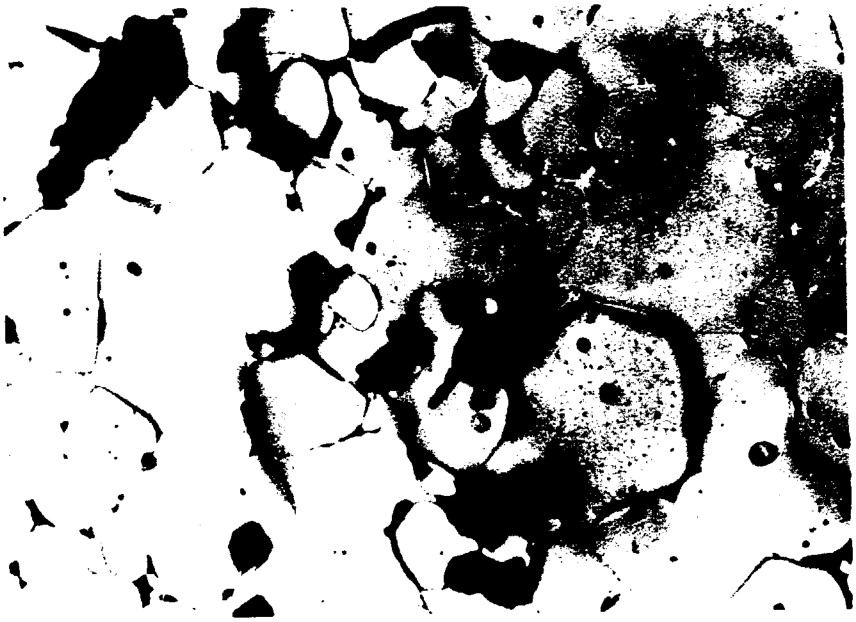

Figure 29. Microphotograph of M-BF magnesite brick Brick matrix is in the middle. Parts of coarse d.b. magnesite grains are left and right; reflected light preparation; enlargement 100 x

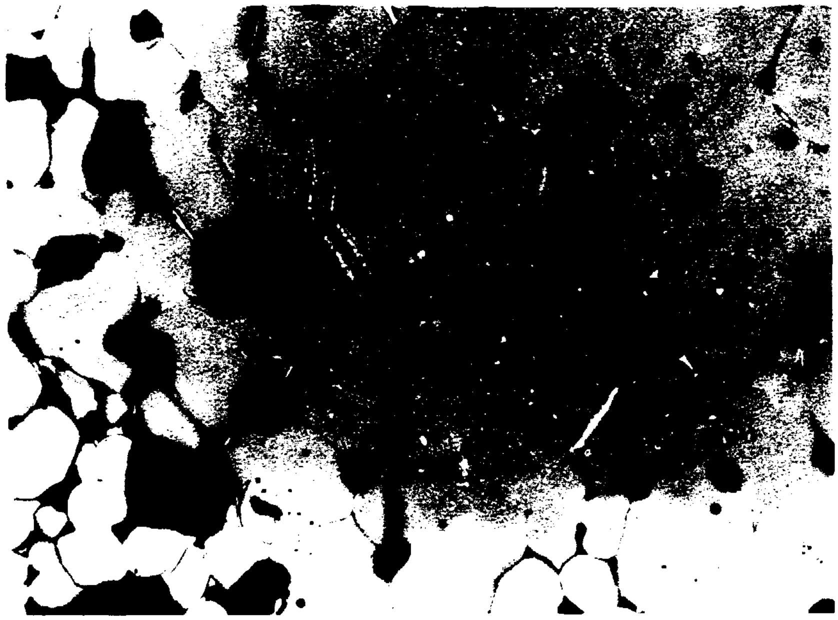

Figure 30. Microphotograph of M-BF magnesite brick. Contact of coarse grained d.b. magnesite (at the left) and brick matrix (at the right) was taken; reflected light preparation; enlargement 100 x

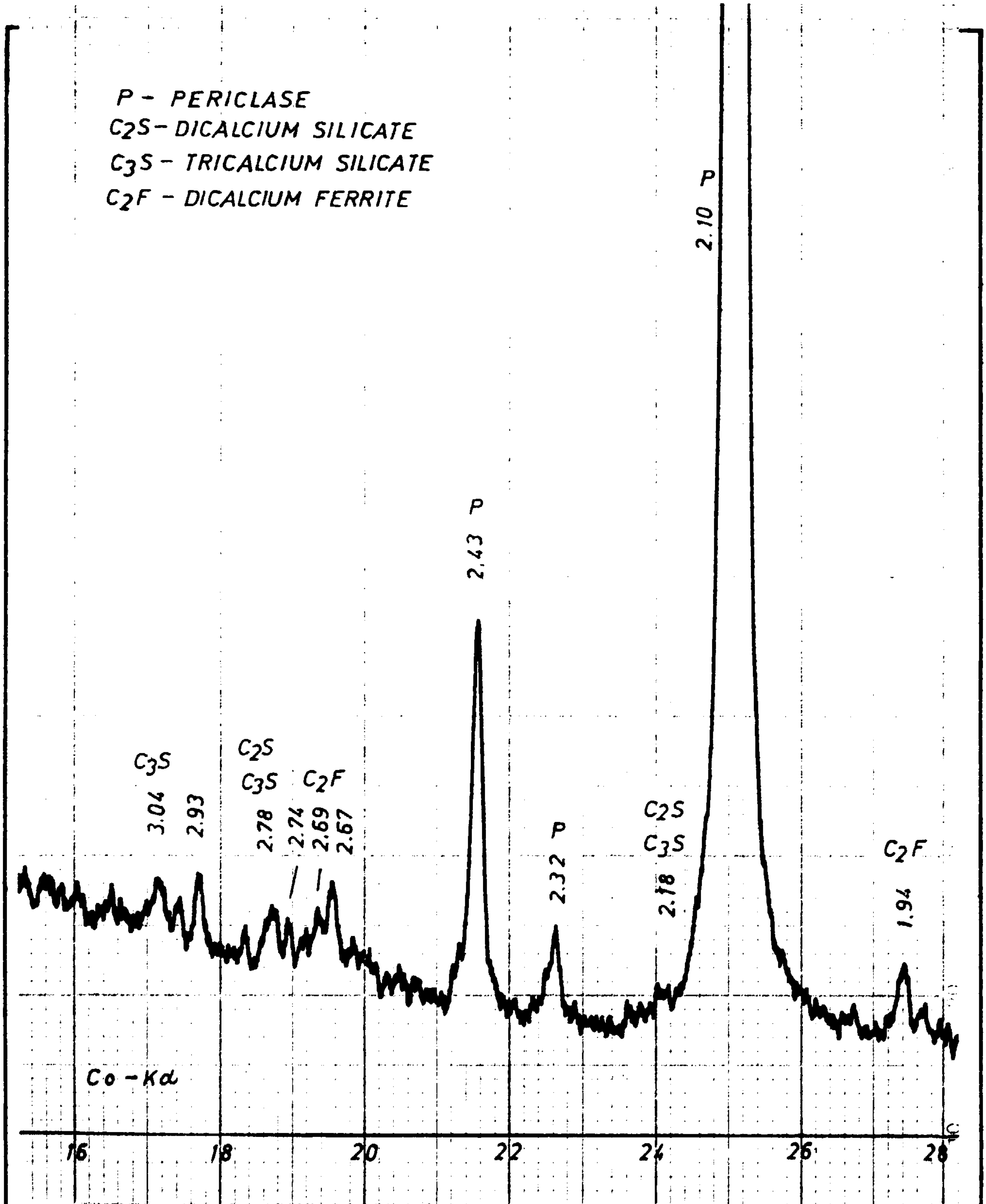

Figure 31. X-Ray pattern of M-BF magnesite brick

M-BS Magnesite brick

Investigations on this brick in polarization microscope showed clearly that there were big differences in appearance of coarse d.b. magnesite grains and of brick matrix. These differences are obvious primarilly because of differences in periclase crystals size in these two brick parts. This caused formation of clearly defined grain structure of brick in which boundaries between coarse d.b. magnesite grains and brick matrix are easily distinguished (figures 32 and 33).

Periclase crystals dimensions in coarse d.b. magnesite grains are 70-90 µm. In brick matrix, crystal dimensions are considerably smaller - about 30 µm.

Investigations have shown that dicalcium silicate was present in the brick, more than in M-A and M-BF bricks. Increase of this silicate content was caused by reaction between quartz and free CaO during DBM-BS d.b. magnesite production. Presence of dicalcium silicate was confirmed by R-Ray diffraction as well (figure 34). Free calcium oxide was not registered.

Comparing to previously described bricks (M-A and M-BF) higher content of pores was found. Pores content is higher in matrix than in coarse d.b. magnesite grains.

In d.b. magnesite grains pore dimensions are from 5 to 120 µm, majority of them being 30 um. Pore dimensions in matrix are 8-300 µm; the largest pores were mightly created during making microscopic preparation. Measurements have shown that majority of pores were about 30 µm.

Bond between periclase crystals was acieved by dicalcium silicate, though with many crystals direct periclase-periclase bond was observed.

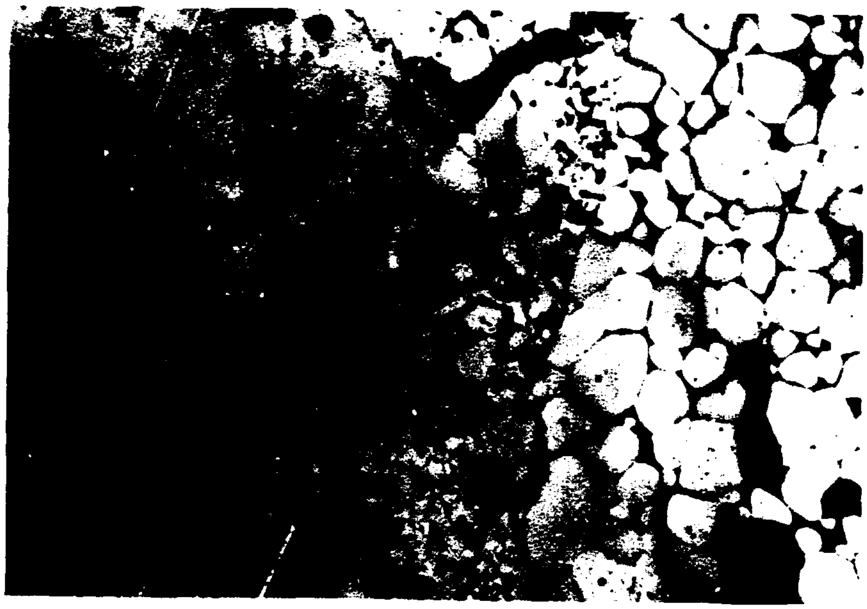

Figure 32. Microphotograph of M-BS magnesite brick. Bri-k matrix is in the middle, coarse d.b. magnesite grains at the left and at the right; reflected light preparation; enlargement 100 x

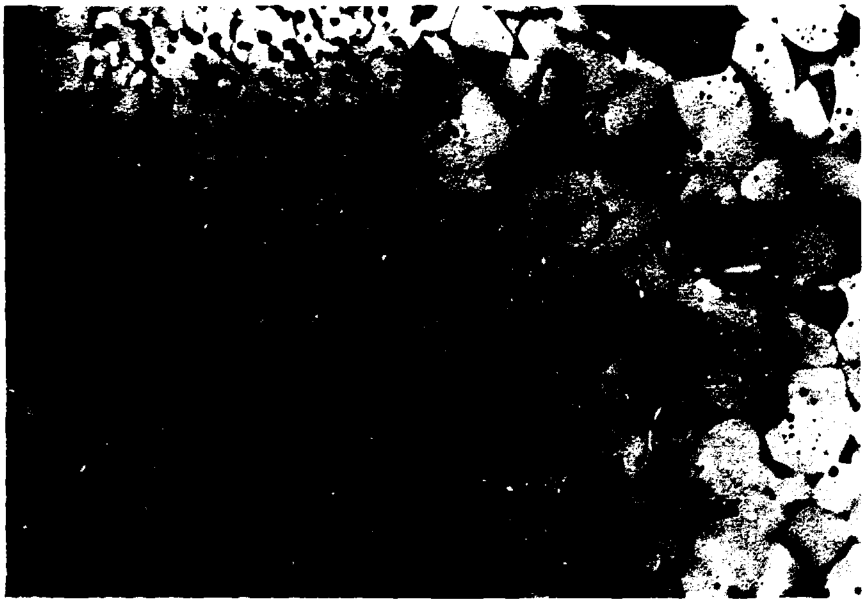

Figure 33. Microphotograph of M-BS magnesite brick. Contact between brick matrix (at the left) and coarse d.b. magnesite grains (at the right) was taken; reflected light preparation; enlargement 100 x

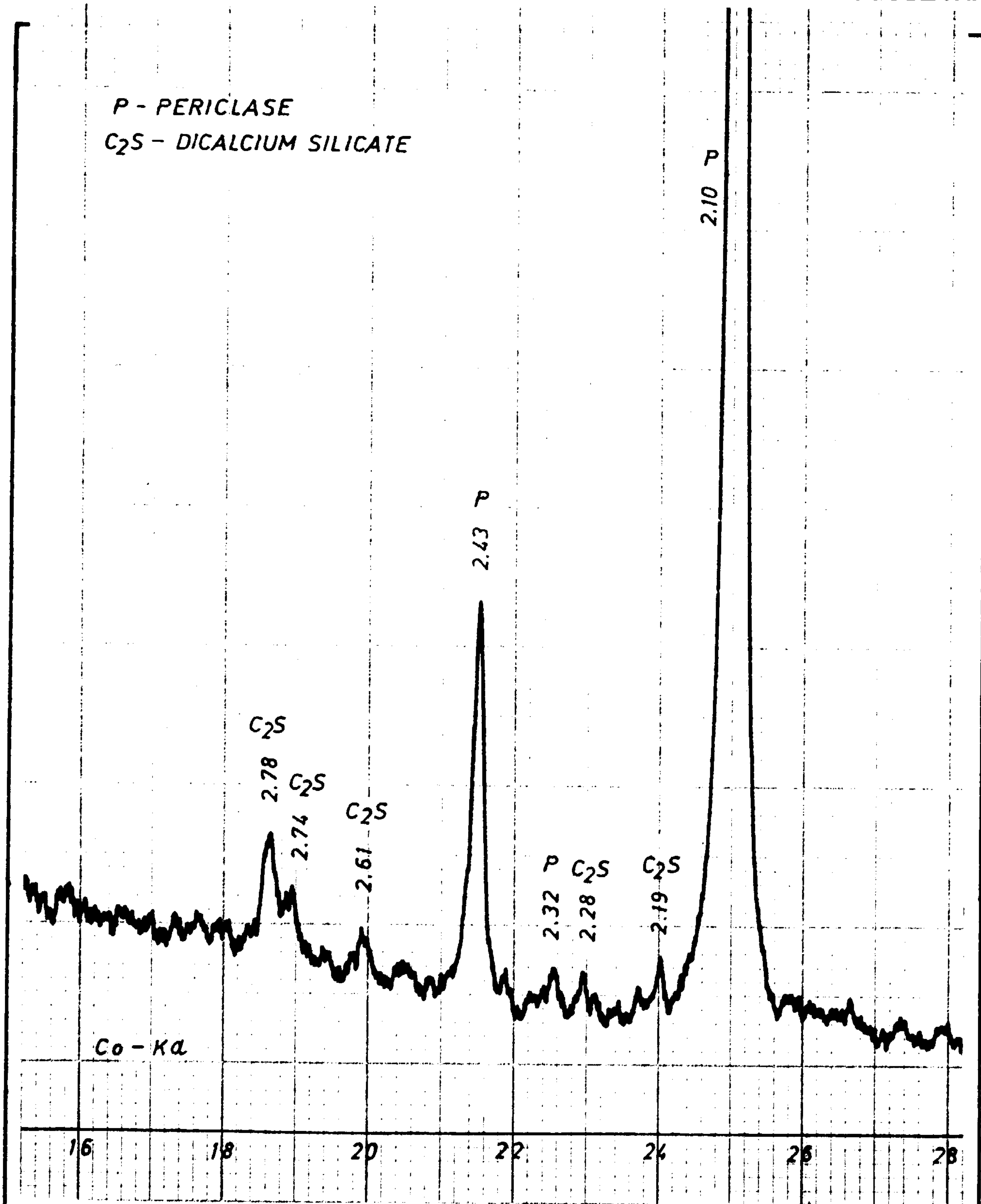

Figure 34. X-Ray pattern of M-BS magnesite brick

C. Discussion of results

1. M-A Magnesite brick

On basis of testing results, one could say that M-A magnesite brick belogs to high grade class of magnesite bricks, regarding its chemical composition, physical properties, mineralogical composition, and microstructure. As it is a brick based on natural d.b. magnesite, M-A brick belogs to a high MgO content class. Interrelations of other oxides are such that provide formation of highly refractory minerals - tricalcium silicates and dicalcijum silicates, which was confirmed by high refractoriness of the brick. The brick has low porosity, good apparent density, satisfactory mechanical characteristics and good thermal shock resistance as magnesite brick. Microstructure of this brick indicates predominant participation of direct bond periclase- periclase, as well as good silicate bond on basis of dicalcium silicate and tricalcium silicate.

By firing the bricks at elevated temperatures, because of high purity of starting d.b. magnesite DBM-A, better values of mechanical characteristics, lower porosity, higher density and bigger periclase crystals were obtained in both d.b. magnesite grains and brick matrix.

2. M-BF Magnesite brick

M-BF magnesite brick could be ranged into medium grade bricks on basis of all testing results. It has an increased content of CaO, successfully stabilized by iron oxide addition. The brick has very good physical properties: low porosity, high apparent density, good mechanical characteristics and excellent thermal shock resistance. Low melting dicalcium ferrite did not notably reduce refractoriness of the brick, because high refractory calcium silicates had bigger effect in bond formation between periclase crystals, as well as considerable effect of direct contact periclase-periclase. In this brick large periclase crystals were obtained both in d.b. magnesite grains and brick matrix, as a result of good recrystalization. On basis of all characteristics achieved in production of this d.b. magnesite DBM-BF based brick we can conclude that all chosen parametes in production process were adequate.

3. M-BS Magnesite brick

On basis of testing results, it can be concluded that MB-S magnesite brick, based on DBM-BS d.b. magnesite, ranges in medium garde bricks, with MgO content that is adequate for this grade of brick and with specially controlled CaO/SiO_2 ratio enabling formation of highly stable dicalcium silicate, in addition to basic mass of periclase. Physical properties are within limits expected for this brick grade. High refractoriness and good mechanical characteristics of the brick are notable. By certain improvements in mixture preparation better porosity could be achieved (16-20%), as well as corresponding increase of apparent density.

Microstructure of this brick indicates that bond between d.b. magnesite grains, as well as betwwen periclase crystals within grains, was mainly based on direct contact periclase-periclase and on highly refractory dicalcium silicate.

Observing investigation results obtained on DBM-B (DBM-BF and DBM-BS) based magnesite bricks, one could say that stabilization of free CaO was successfully achieved in both cases. However, each method of stabilization has its advantages reflecting on brick characteristics. Having in mind requirements for high refractoriness, good thermal shock resistance and good mechanical characteristics of bricks we think that best bricks within this grade could be obtained by combined stabilization of CaO in d.b. magnesite with addition of quartz and iron oxide.

VII. PRODUCTION OF TEST MAGNESITE-CHROME BRICKS MC-A

A. Production

Dead burned magnesite, grade DBM-A, was used for production of bricks with chromite. Because of limited quantity of d.b. magnesite, only one grade of magnesite-chrome bricks was produced, containing about 15% of Cr_2O_3, and the following components were used:

- d.b. magnesite DBM-A
- chrome ore CF 50-200
- chrome concentrate CCD26.

1. Preparation of components and their characteristics

Granular componnets of d.b. magnesite were prepared in laboratory, through crushers system with upper limit size of 4 mm, and chrome ore was comminuted in jaw crushers and roll mill with upper limit size of 3 mm. Chrome concentrate was used without additional tretment. Fine component of d.b. magnesite was prepared in laboratory ball mill.

Chemical composition of components is given in table No. 19.

Table 19.
Chemical composition of components, in %

	Ign. loss	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Cr_2O_3
D.b.magnesite DBM-A	0.23	0.68	0.12	0.20	2.62	96.13	
Chrome ore CF 50-200	1.62	4.15	9.65	18.13	0.37	13.52	52.56
Chrome concentrate CCD26	0.14	1.02	11.10	15.40	0.24	15.14	56.96

2. Production of bricks

With 70% of dead burned magnesite and 30% of chromite component, and with necessary participation of separate classes of d.b. magnesite and chromite, the brick mixture was composed.

The mixture was prepared according to the usual procedure, so that granular components of d.b. magnesite and chromite were first dosed into the mixer, and then binding material and, finally, fine component. After completion of homogenization, bricks were pressed on oil-hydraulic press in standard shape "2" under the pressure of 100 N/mm^2. After drying during 24 hours on adequate temperature, the bricks were fired in industrial tunnel kiln at the maximum firing temperature of 1720°C.

During firing slight shrinkage occured of all dimensions, not exceeding the limits allowed by standards. Fired brick is of good macroscopic appearance , has sharp edges and smooth surfaces.

B. Investigation of bricks

Chemical, thermophysical and mineralogical investigations were done on fired bricks.

1. Chemical and thermophysical characteristics

Chemical composition and thermophysical characteristics . presented in table 20. Physical characteristics represent the average of at least two measurements.

Table 20.

Chemical composition and thermophysical characteristics of MC-A bricks

Apparent porosity, %	17.54
Apparent density, g/cm^3	3.04
Refractoriness under load, Ta, ^0C	+ 1700
Crushing strength at 20^0C, N/mm^2	37
Crushing strength at 1400^oC, N/mm^2	38
Modulus of ruptire (MoR) at 1400^oC, N/mm^2	14
Linera thermal expansion up to 1400^oC, %	1.5
Thermal shock resistance in air (change)	+ 30
Ignition loss, %	0.12
SiO_2, %	1.24
Al_2O_3	3.32
Fe_2O_3, %	5.64
CaO, %	2.21
MgO, %	70.50
Cr_2O_3, %	16.97

2. Mineralogical investigations of MC-A bricks

In orded to observe structural characteristics and to record mineral phases in brick, microscopic and X-Ray investigations were done.

In microscopic preparation of this brick microstructural characteristics are clearly observed indicating that during firing intensive reactions between periclase crystals and chromite occured.

These reactions were particularly intensive between chromite concentrate and fine particles of d.b. magnesite in brick matrix. In that way quality matrix was formed comprising all three sorts of bonds between periclase crystals, as well as between periclase nad chromite, i.e. cilicate bond, direct bond (periclase-periclase nad periclase-chormite) and bond by secondary spinelides (figure and 36).

Silicates in matrix are mainly present as dicalcium silicates, though in some parts of brick a silicate is present which by its optical characteristics resambles mervinite. Mervinite was formed by reaction between dicalcium silicate and magnesite hydrosilicates, entered in brick mass with chromite. Small particles of chromite in brick matrix reacted with periclase or silicates to a great extent. Because of these reactions comparatively big quantity of secondary spinelides in form of spoty inclusions was formed; in silicates these were crystals often of more regular shapes. Secondary spinelides in silicates represent an additional high refractory phase between periclase crystals. Chromite-periclase reactions caused formation of direct periclase-periclase bond, particularly periclase-chromite.

During firing some changes occured in coarse d.b. magnesite grains. Periclase crystals in these grains , near chromite, were enriched with considerabla quantity of inclusions os secondary spinelides. Due to these reactions at high temperatures, recrystallization of periclase took place, with crystals are often over 100 μm big. In coarse grains of d.b. magnesite high level of direct periclase-periclase bond was observed. In these d.b. magnesite grains, dicalcium silicate is mainly present, but in smaller quantity compared to brick matrix. Bond between coarse d.b. magnesite grains and brick matrix was mailny achieved by silicates and by direct periclase-periclase bonds.

In microscopic preparations, high extent of direct bond was also observed between coarse chromite grains and periclase crystals (figure 36)

Pores are present in much bigger qualntity in brick matrix than in coarse d.b. magnesite grains.

By X-Ray examinations presence of periclase and chromite was stated (figure 37). Silicates could not be registered for sure because due to presence of chromite, increase of background occured preventing better registering of difraction reflections of silicates.

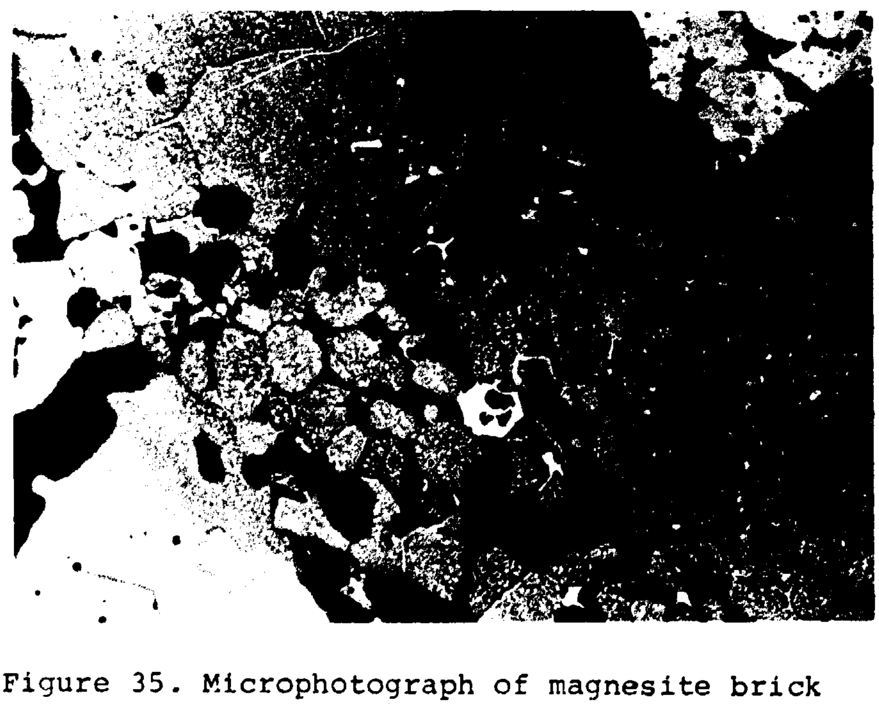

Figure 35. Microphotograph of magnesite brick MC-A. Brick matrix is in the middle. Reflected light preparation; enlargement 100 x

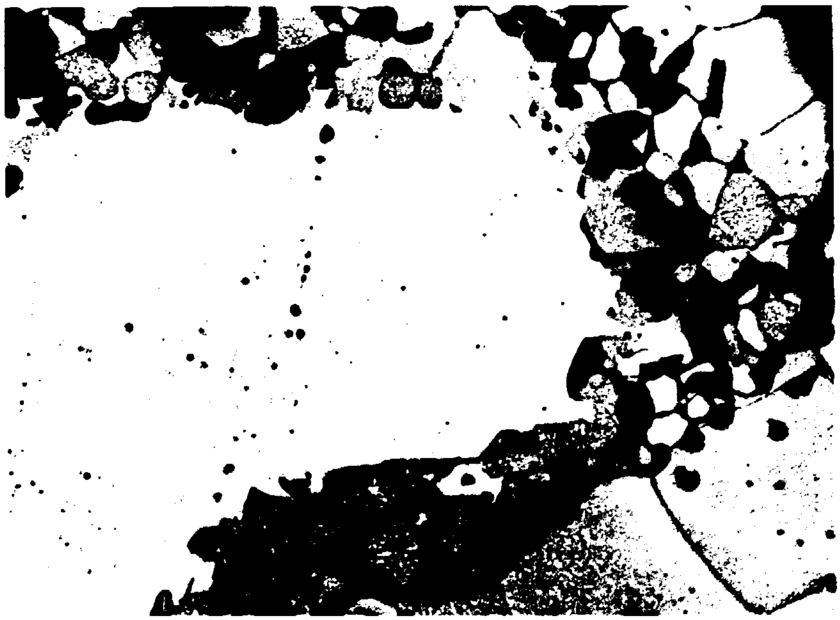

Figure 36. Microphotograph of magnesite brick MC-A. Coarse chromite grains are in the middle and in the left corner; Reflected light preparation; enlargement 100 x

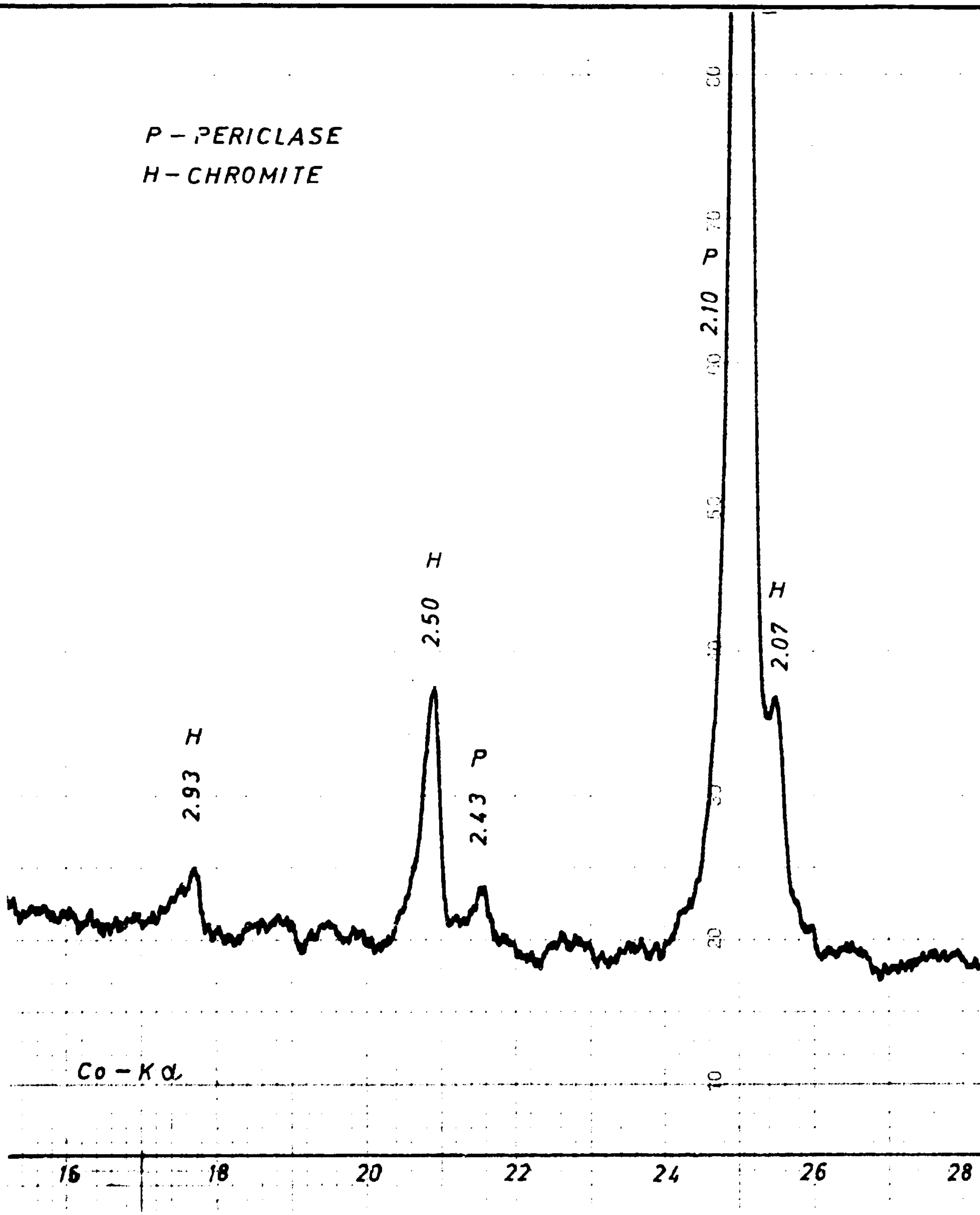

Figure 37. X-Ray pattern of magnesite-chrome brick MC-A

C. Discussion of results

Magnesite-chrome brick, grade MC-A, was produced of natural dead burned magnesite with very low SiO_2 content and C/S ratio over 3. Chromite ore was of usual refractory type with high content of Cr_2O_3, while chromite concentrate was also with very low SiO_2 content and high Cr_2O_3 content. Beginning with such pure raw materials, by firing at high temperature, quality magnesite-chrome brick was obtained with low porosity and good density. Very good mechanical characteristics at elevated temperatures should be separately pointed out, such as: crushing strength and modulus of rupture at $1400^{o}C$.

By microscopic investigation, presence of direct bonds periclase-periclase and periclase-chromespinelide were identified. Silicates were presented in forms of dicalcium silicate and mervinite, which, as highly refractory minerals, together with secondary spinelides, gives good characteristics at elevated temperatures.

Considering obtained results of chemical and mineralogical composition, structural characteristics and thermophysical characteristics, it could be said that test bricks were produced of very good quality.

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ANNEX No. 1

PROGRAM

LABORATORY INVESTIGATIONS ON POSSIBILITY OF OBTAINING HIGH GRADE TEST DEAD BURNED MAGNESITE, MAGNESITE BRICKS AND MAGNESITE-CHROME BRICKS OF UPGRADED CALCINED ZIMBABWEAN MAGNESITE

1. DETERMINATION OF CHEMICAL AND MINERALOGICAL COMPOSITIONS OD SEMI-PRODUCTS: SP-A AND SP-B

2. MILLING OF SEMI-PRODUCTS

2.1. Determination of characteristics of fine milled semi-products
   - Grain size distribution
   - Packing density
   - Specific surface

3. BRIQUETTING

3.1. Selection of conditions for dense briquettes
   - Pre-compacting
   - Velocity of rollers
   - Pressure
   - Temperature of material

3.2. Determination of characteristics of briquettes obtained
   - Sterngth
   - Apparent density
   - Packing density

4. FIRING OF BRIQUETTES

4.1 Firing schedule determination
   - Duration: 2 hours
   - Temperatures: 1700, 1800 and 1900 $^{\circ}C$

4.2. Determination of characteristics of fired briquettes

- Chemical composition
- Mineralogical investigation
  - a) Mineralogical composition
  - b) Microstructure
  - c) Periclase crystals size
- Physical properties
  - a) Density
  - b) Apparent density
  - c) Porosity
  - d) Hydration resistance

5. PRODUCTION OF TEST MAGNESITE BRICKS OF DEAD BURNED MAGNESITE: DBM-A, DBM-BF AND DBM-BS

5.1. Preparation of components

5.2. Preparation of mixtures for bricks pressing

5.3. Pressing of test bricks

5.4 D r y i n g

5.5. Firing of test bricks

5.6. Determination of characteristics of test bricks

- Macroscopic appearance
- Chemical composition
- Mineralogical investigations
  - a) Mineralogical composition
  - b) Microstructure
- Physical properties
  - a) Crushing strength
  - b) Apparent density
  - c) D e n s i t y
  - d) Apparent porosity
  - e) Distribution of pores
  - f) Refractoriness under load (Ta)
  - g) Thermal shock resistance (Tw)
  - h) Thermal expansion, 20-1400 ^{o}C

6. PRODUCTION OF TEST MAGNESITE-CHROME BRICKS OF DEAD BURNED MAGNESITE: DBM-A

6.1. Preparation of components

6.2. Preparation of mixture for bricks pressing

6.3. Pressing of test bricks

6.4 Drying of test bricks

6.5. Firing of test bricks

6.6. Determination of characteristics of test bricks

- Macroscopic appearance
- Chemical composition
- Mineralogical investigations
  a) Mineralogical composition
  b) Microstructure
- Physical properties
  a) Crushing strength
  b) Apparent density
  c) D e n s i t y
  d) Apparent porosity
  e) Distribution of pores
  f) Refractoriness under load (Ta)
  g) Thermal shock resistance (Tw)
  h) Thermal expansion, 20-1400°C

7. CONCLUSION

8. RECCOMANDATION FOR FURTHER WORK ON REALIZATION OF THE PROJECT: BASIC REFRACTORIES PLANT IN ZIMBABWE

Time required: Five (5) months after signing of the Contract.

Annex No. 2

PROPOSED PROGRAM OF SEMI-INDUSTRIAL INVESTIGATIONS

PART I. ACTIVITIES TO BE ACHIEVED IN REPUBLIC OF ZIMBABWE

1. Taking of representative samples

1.1. Taking of the representative samples of raw magnesite (18 t) from Kadoma Mine:

a) 6 t of raw magnesite (3-25 mm)
6 t of raw magnesite (3-60 mm)
6 t of raw magnesite (25-60 mm)

b) Hand selection and separation of dark ore pieces

c) Transportation to Eiffel Flats, near Kadoma

1.2. Taking samples of chrome ore and of chrome concentrate:

a) 1.5 t of lumpy chrome ore from Frances Mine, Zimbabwe (50-200 mm)
In case that lumpy chrome ore from Frances Mine could not be obtained, substitution with coarse chrome concentrate designated as "D-20" from Mtoroshanga Mine, Zimbabwe, is acceptable.

b) 2 t of chrome concentrate designated with "D-26" from Mtoroshanga Chrome Mine, Zimbabwe

c) 0.75 t of flake graphite from Graphite Mines (PVT) Limited, Harare

2. Determination of representative samples characteristics:

a) Grain size distribution
b) Chemical composition (E. Flats, DoM, IMR)
c) Mineralogical composition (IMR)
d) Differential Thermal Analysis (DTA) (DoM)

E. Flats - Eiffel Flats
DoM - Dep. of Metallurgy, Harare
IMR - Institute of Mining Research

2.1. Evaluation of the results obtained
Place of realization: DoM, Harare

3. Calcining test in semi-industrial rotary kiln (Eiffel Flats, Kadoma) - PREVIOUS INVESTIGATIONS.

Adaption of the existing kiln, 10.5 m of length, Ø 770/922 mm and lining with refractory bricks in a manner to ensure reliable results of calcining test, counting on operating tepmeratures up to 1200°C.

It is also needed to provide all necessary equipment listed in Annex No. 3, prior to beginning of activities.

Acceptable fuel is light diesel oil or earth gas, depending on which one is easier and cheaper to provide in Republic of Zimbabwe. Adequate burner is to be provided with needed installations and reservoir of corresponding volume - also prior to beginning of activities on investigations.

3.1. Putting the rotary kiln in operation

- Testing in cold state
- Heating up the rotary kiln

3.2. First calcining tests are to be performed with all three different fractions of raw magensite, i.e.:

3-25 mm, 3-60 mm and 25-60 mm

During these tests the following factors effecting the quality of raw magnesite are to be determined:
- effect of material size
- effect of operating kiln temperatures, 900 - 1200°C

3.3 Determination of calcined magnesite characteristics:
- Grain size distribution
- Chemical composition
- DTA analyses
- Reactivity with water

3.4. Determination of optimum conditions for beneficiation process by selective hydration procedure (wetting and soaking).

3.5. Evaluation of results, selection of optimum conditions for calcining process and selection of optimum conditions for beneficiation process.

Place of realization: Kadoma

4. Collecting representative sample for production of calcined magnesite. Quantity of raw magnesite that is to be taken from the mine is 140 t. - MAIN TEST.

4.1. Transportation of representative sample from Kadoma Mine to semi-industrial rotary kiln in Eiffel Flats, near Kadoma.

Characteristics determination of representative sample, 140 t.
- Grain size distribution (E. Flats)
- Chemical composition (E. Flats, Dom, IMR)

4.3. Evaluation of results

Place of realization: Kadoma

5. Production about 55 t of calcined magnesite for beneficiation process - MAIN TEST:

5.1- Regular checking of calcining process:

- Chemical composition (every 4 hours)(E. Flats)
- Grain size distribution (every 4 hours (E. Flats)
- Reactivity of calcined material (once per shift) (E. Flats)

5.2. Determination of characteristics of calcined magnesite for beneficiation process - Composite sample:

- Grain size distribution (DoM)
- Chemical composition (E. Flats, Dom. IMR)
- Reactivity with water (DoM)
- Mineralogical composition (IMR)
- DTA analysis (DoM)

5.3. Determination of specific consumption values for calcining process:

- Raw magne ite
- Fuel
- Electric energy for work of rotary kiln

Place of realization: Kadoma

6. Beneficiation of calcined magnesite by selective hydration procedure - MAIN TEST

6.1. Transportation about 55 t of calcined magensite to Harare (DoM)

6.2. Production of about 20-22 t of semi-product (fraction "A") and about 16-18 t of semi-product (fraction "B").

6.3. Control of the optimum conditions for beneficiation process by procedure of selective hydration (wetting and soaking)

6.4. Determination of semi-products characteristics (fractions "A" and "B"):

- Grain size distribution (DoM)
- Chemical composition (E. Flats, DoM, IMR)
- Mineralogical composition (IMR)
- DTA analysis (DoM)
- Filling mass

6.5. Determination of specific consumption values for beneficiation process:

- Calcined magnesite
- Water
- Electric energy

7. Evaluation of results for main test

8. Conclusions and recommandations

9. Final Report for the activities performed in Republic of Zimbabwe (10 copies)

Place of realization: DoM, Harare

PROPOSED PROGRAM OF SEMI-INDUSTRIAL INVESTIGATIONS

PART II: ACTIVITIES TO BE ACHIEVED IN YUGOSLAVIA/EUROPE

1. Fine milling 20-22 t of semi-product (fraction "A")

1.1. Selection of mill and of condition for its operation

1.2. Determination of characteristics of finely milled semi-product (fraction "A"):
- Grain size distribution
- Filling mass

1.3. Evaluation of resuts of finely milling process

2. Production of dense briquettes, 13-15 t

2.1. Selection of working conditions for briquetting process

2.2. Determination of "green" briquettes characteristics:
- Bulk density
- Filling mass

2.3. Determination of specific consumption values:
- Finelly milled semi-product (fraction "A")
- Electric energy

2.4. Evaluation of all results of briquetting process

3. Dead burning of briquettes in semi-industrial kiln

3.1. Selection of working conditions for the dead burning process

3.2. Regular control of dead burning process
- Chemical composition
- Bulk density

3.3. Production of high grade dead burned magnesite, 10-12 t

3.4. Determination of cpecific consumption values:
- Briquettes
- Fuel
- Electric energy

3.5. Evaluation of all results of dead burning process

4. Production of magnesite bricks, periclase-carbon bricks and magnesite-chrome test bricks, 10-12 t, total

4.1. Determination of characteristics of high grade dead burned magnesite:
- Chemical composition
- Mineralogical composition
- Microstructure
- Size of periclase crystals
- Physical properties
  - Density
  - Bulk density
  - Porosity
  - Grain bulk density
  - Hydration resistance

4.2. Preparation of components for brick production and mixing
4.3. Pressing of test bricks
4.4 Drying and tempering of test bricks
4.5. Firing of test bricks
4.6. Determination of characteristics of magnesite test bricks, periclase-carbon test bricks and magnesite-chrome test bricks:
- Macroscopic appearance
- Chemical composition
- Mineralogical composition
- Cold crushing strength
- Apparent porosity
- Refractoriness under load
- Thermal shock resistance
- Linear expansion coefficient

4.7. Determination of specific consumption values
- Raw materials
- Fuel
- Electric energy

4.8. Evaluation of all results
5. Conclusions and recommandations
6. Elaboration of Final Report about acivities executed in Yugoslavia/Europe (10 copies).

ANNEX No. 3

LIST OF THE EQUIPMENT WHICH IS TO BE PROVIDED FOR WORK ON SEMI-INDUSTRIAL INVESTIGATIONS IN REPUBLIC OF ZIMBABWE

1. Rubber Belt Conveyor that can also be movable. Intended for feeding semi-industrial rotyra kiln with raw magnesite. More detailled description of this kiln is given as item No. 2 of this list.

2. Semi-Industrial Rotary Kiln for the forseen semi-industrial investigations; the existing kiln in Eiffel Flats (under a roof) could be used (10400 mm of length). The kiln has to be adapted and mechanically repared, as well as lined with refractory bricks of adequate quality, as to reach working temperatures over 1200°C.

   For this kiln adequate burner is to be provided to use light diesel oil or earth gas, enabling proper calcination of raw magnesite up to 1200°C, according to the kiln capacity.

   Prior to beginning of works, the kiln is to be mechanically tested in cold state.

3. Optical Temperature Measuring Instrument to control working temperature in rotary kiln during calcining. Its measuring range should be approximately 700-1300°C, with accuracy of ± 10°C. The instrument is to enable the whole quantity of raw magnesite to be as evenly calcined as possible, as well as to enable temperature control at different levels.

4. Mixer for Wetting the Calcined Magnesite

   For this process mobile mixer could be used, like the one used for mixing concrete in civil engineering. It should be about 100 liters of volume, or close of that volume, obtainable at the market - from regular serial production.

5. Containers made of zink plated sheet, about 75 liters of volume (20 pcs). The containers are intended for taking over the calcined magnesite after wetting in concrete mixer. The 20 pcs are to enable undisturbed work for 2 - 3 days.

6. Vybrating Screen, double decked, with capacity of 200-250 kgs/h. As no special requests are made regarding construction of the screen itself (it is only desirable that it should be dedusted), an ordinary screen from regular production could be used, having about the required capacity, which can be found in Zimbabwe. Mesh sizes of the screens should be as follows: a) the upper screen 3 x 3 mm; b) the lower screen 1 x 1 mm. The screens used in civil engineering could be used for this purpose.

7. Cold Crushing Tester with the Attachment for pressing the samples tested

   The tester is indispensable for development of new products in fine and coarse ceramics and constructional materials. It would considerably facilitate work and extend activities of Department of Metallurgy. An Offer of the best known European producer was acquired (TONI-MEL Prufsysteme, D-6707 Schifferstadt, No. 1982/85/0 of 16th October 1985). The copy of the Offer was given to Zimbabwean partner