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(54) **Title:** COMPOSITION SUITABLE FOR HOT REPAIRING REFRACTORIES

(57) **Abstract:** The present invention relates to a composition [composition (C), herein-after] suitable for hot repairing refractories, comprising a particulate mixture consisting of at least one first fraction consisting of first particles having a first chemical composition and at least one second fraction consisting of second particles having a second chemical composition, wherein the first fraction has a first d_{50} particle size value between 0.5 - 10 mm and the at least one second fraction has a second d_{50} particle size value which is wherein the ratio between the second d_{50} particle size value of the second fraction and the first d_{50} particle size value of the first fraction is from 0.05 to 0.95, and wherein the first chemical composition, relative to the total weight of the first chemical composition comprises: - from 20.0 wt.% to 45.0 wt.% of at least one first magnesium oxide compound, as expressed in wt.% of MgO; - from 40.0 wt.% to 60.0 wt.% of at least one calcium oxide compound, as expressed in wt.% of CaO; - from 5.0 wt.% to 12.0 wt.% of at least one iron oxide compound, as expressed in wt.% of Fe_2O_3, and wherein the second chemical composition comprises at least one second magnesium oxide compound, wherein the total content of the first and the second magnesium oxide compounds in the particulate mixture, relative to the total weight of the particulate mixture is from 30.0 to 80.0 wt.%, as expressed in wt.% of MgO. The invention further relates to methods for manufacturing of the composition (C), and to method for hot-repairing a refractory lining (L) wherein said refractory lining (L) is hot-repaired with the composition (C).

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COMPOSITION SUITABLE FOR HOT REPAIRING REFRACTORIES

FIELD OF THE INVENTION

The present invention relates to a composition (C) suitable for hot repairing refractories. The present invention further relates to a method for manufacturing said composition (C), to a method for hot-repairing a refractory lining (L) and to a sintered hot repair material obtained by said method for hot-repairing a refractory lining (L) wherein said refractory lining (L) are treated with the composition (C).

BACKGROUND OF THE INVENTION

The lining of the inner wall of vessels (notably Electric Arc Furnace – EAF) with refractories is essential for protecting vessels during iron and steel-making processes. Due to their direct contact with the molten metals, refractory linings are subjected to high temperature and corrosive environment. These extreme conditions inevitably lead to the wear out of the refractory linings through corrosion and abrasion. The refractory lining hence needs to be dismantled and replaced periodically.

In order to prevent early replacement of refractory linings, it is for example known to repair them, by notably applying additional refractory materials, called hot-repair materials, onto refractory linings. Sacrificial layers are consequently created when the vessel is reused for a subsequent steel-making process. The sacrificial layers hinder or even prevent the dissolution of the refractory linings. It is important that the hot repair material is compatible with the refractory lining. Consequently, the chemical composition of hot repair materials needs to be tailored to the chemical composition of the refractory brick lining.

Furthermore, it is important that the hot repair materials show a good flowability. Indeed, hot repair materials are typically fettled onto the refractory lining. The flowability, and more in particular the processability, the transport and the storage of the hot repair materials, is therefore an important parameter for a good execution of hot-repairing refractories.

Magnesia-based materials are known to be used as hot repair materials. They are characterized by having a high magnesium oxide (MgO) content. However, their high cost and the scarcity of high purity magnesium oxide makes their use as hot-repair materials at an industrial scale less interesting.

As an alternative to magnesia-based materials, F. Firsbach *et al.* (Innovative refractories for iron and steelmaking, International Colloquium on Refractories - ICR®,113-118) describe the use of a dolime-based material with iron ore coating (Dolofrit® 180) for hot repairing the refractory brick lining of Electric Arc Furnaces (EAFs). Such material requires the use of high purity dead-burnt dolime based material.

In order to address the high manufacturing cost of high purity magnesia as raw material for hot repair, efforts have been made to recycle waste materials originating from iron and steel-making processes. Indeed, such processes generate substantial amounts of wastes or by-products. Due to the high amount of wastes generated and with evident concern on the environmental and economic impact of such wastes, recycling waste materials has been a longstanding issue in the iron and steel industry. To this end, it is for example known from M.A. Serry *et al.* (Refractories Manual 2003, 46-50) to use recycled magnesite brick powder as raw material. Along with mill scale, and dolomitic-magnesite and/or dolomite raw materials, recycled magnesite brick powder is incorporated to provide co-clinkered grains as hot-repair material.

This document further discloses that the co-clinkered grains were manufactured by mixing the raw materials, as disclosed above, in powder form. In subsequent steps, the resulting mixed powder was notably clinkered, crushed, graded and mixed, thereby providing said co-clinkered grains. The clinkering step, involving heating said mixed powder at high temperature, therefore allows the different components to chemically react. However, a clinkering step requires high energy consumption.

As an alternative of using hot-repair materials, US 2,943,240 describes a dolomite-containing refractory lining material for use in furnaces, with the aim to provide a refractory lining material having an extended storage life, and notably having a good resistance for an extended period of time. In particular, this reference teaches that refractory lining bricks comprising dolomite-containing lining materials have improved erosion resistance.

In view of all the above, there remains a need for improved compositions suitable for hot-repairing refractories, which, when applied to refractory linings of a vessel, provide an improved refractory lining resistance against corrosion, thereby preventing the early replacement of refractory linings. There remains also a need for compositions which have a good flowability and more in particular a good processability, transportation and storage, while contributing to sustainability by providing a hot-repair material which is part of circular economy and shows excellent quarry life.

SUMMARY OF THE INVENTION

The inventors have now surprisingly found that it is possible to provide an improved composition, suitable for hot repairing refractories, fulfilling the above-mentioned needs.

Thus, there is now provided a composition [composition (C), herein-after] suitable for hot repairing refractories, comprising a particulate mixture consisting of at least one first fraction consisting of first particles having a first chemical composition and at least one second fraction consisting of second particles having a second chemical composition, wherein the first fraction has a first d_{50} particle size value between 0.5 – 10 mm and the at least one second fraction has a second d_{50} particle size value , wherein the ratio between the second d_{50} particle size value of the second fraction and the first d_{50} particle size value of the first fraction is from 0.05 to 0.95, and

wherein the first chemical composition, relative to the total weight of the first chemical composition comprises:

- from 20.0 wt.% to 45.0 wt.% of at least one first magnesium oxide compound, as expressed in wt.% of MgO,
- from 40.0 wt.% to 60.0 wt.% of at least one calcium oxide compound, as expressed in wt.% of CaO
- from 5.0 wt.% to 12.0 wt.% of at least one iron oxide compound, as expressed in wt.% of Fe_2O_3, and

wherein the second chemical composition comprises at least one second magnesium oxide compound, wherein the total content of the first and the second magnesium oxide compounds in the particulate mixture, relative to the total weight of the particulate mixture is from 30.0 to 80.0 wt.%, as expressed in wt.% of MgO.

In another aspect, the present invention further provides a composition (C) suitable for hot repairing refractories, comprising a particulate mixture consisting of at least one first fraction consisting of first particles and at least one second fraction consisting of second particles, wherein the first fraction has a first d_{50} particle size value between 0.5 – 10 mm and the second fraction has a second d_{50} particle size

value wherein the ratio between the second d_{50} particle size value of the second fraction and the first d_{50} particle size value of the first fraction is from 0.05 to 0.95, and wherein said first fraction has the second chemical composition as described herein-above,
and wherein said second fraction has the first chemical composition, as described herein-above.

In another aspect, the present invention further provides a method for the manufacturing of the composition (C) as detailed above.

In another aspect, the present invention further provides a method for hot-repairing a refractory lining [refractory lining (L) herein-after], wherein the refractory lining is hot-repaired with the composition (C), as detailed above and wherein the composition (C) is applied to the refractory lining (L).

In another aspect, the present invention further provides a sintered hot repair-material obtained by the method for hot-repairing refractories, as detailed above, wherein said refractories are treated with the composition (C), as detailed above.

DETAILED DESCRIPTION OF THE INVENTION

Within the context of the present invention, the term “comprising” should not be interpreted as being restricted to the means listed thereafter; it does not exclude other elements or steps. It needs to be interpreted as specifying the presence of the stated features, integers, steps or components as referred to, but does not preclude the presence or addition of one or more other features, integers, steps or components, or groups thereof. Thus, the scope of the expression “a composition comprising components A and B” should not be limited to compositions consisting only of components A and B. It means that with respect to the present invention, the only relevant components of the composition are A and B. Accordingly, the terms “comprising” and “including” encompass the more restrictive terms “consisting essentially of” and “consisting of”.

As detailed herein-above, the composition (C) comprises a particulate mixture consisting of at least one first fraction consisting of first particles having a first chemical composition, as detailed above, and at least one second fraction consisting of second particles having a second chemical composition, as detailed above, wherein the at least one first fraction has a first d_{50} particle size value between 0.5 –

10 mm and the at least one second fraction has a second d_{50} particle size value wherein the ratio between the second d_{50} particle size value of the second fraction and the first d_{50} particle size value of the first fraction is from 0.05 to 0.95.

Within the context of the present invention, the expressions “at least one first fraction” and “at least one second fraction” are intended to denote one first fraction or more than one first fraction, and one second fraction or more than one second fraction, respectively.

In the rest of the text, the expressions “first fraction”, and “second fraction” are understood, for the purpose of the present invention, both in the plural and in the singular form, that is to say that the composition (C) of the present invention comprising a particulate mixture consisting of one first fraction or more than one first fraction, and one second fraction or more than one second fraction, respectively.

Within the context of the present invention, the expression “the first fraction has a first d_{50} particle size value between 0.5 – 10 mm and the at least one second fraction has a second d_{50} particle size value wherein the ratio between the second d_{50} particle size value of the second fraction and the first d_{50} particle size value of the first fraction is from 0.05 to 0.95” refers either to the first d_{50} particle size value, and to the second d_{50} particle size value, respectively, when the composition (C) comprises the particulate mixture consisting only of one first fraction and one second fraction, or to each of the first d_{50} particle size value, and to each of the second d_{50} particle size value, respectively, when the composition (C) comprises the particulate mixture consist of more than one first fraction and more than one second fraction.

The Inventors have surprisingly found that by using a particulate mixture, as detailed above, consisting of a first fraction, as detailed above, combined with a second fraction, as detailed above, the composition (C) thereby obtained possess excellent porosity, density and sintering properties. Consequently, when the composition (C) according to the present invention is used for hot-repairing refractories, its resistance to slag corrosion is improved, as demonstrated in the working examples. Furthermore, the inventors have found that by using the particulate mixture, as detailed above, the shrinkage of the composition (C), is influenced, as demonstrated in the working examples.

Within the context of the present invention, the expression « d_x of y mm » and “d_x of y µm” is intended to denote that a percentage (x%) by weight of particles has a particle size equal to or less than y mm and a percentage (x%) by weight of particles has a particle size equal to or less than y µm, respectively.

In general, the particle size distribution of particles can be measured by known method in the art such as Dynamic Light Scattering (DLS), dry or wet mechanical sieving, or laser diffraction analysis.

Preferably, the particle size distribution of the particles of the first and second fraction of the composition (C) is measured by mechanical sieving according to EN ISO 1927-3 (2013) standard or by laser diffraction analysis.

More preferably, the particle size distribution of the particles of the second fraction of the composition (C) is measured by laser diffraction analysis.

In general, mechanical sieving measurement is based on the mechanical separation of a fraction (*i.e.* each of the first fraction and second fraction of the composition (C)) on a series of superimposed sieves. In general, mechanical sieving measurements is determined with an automated mechanical sieving device. Said automatic sieving device comprises means for mounting a nest of sieves, which is located on a vibrating sieve shaker. The sieves are superimposed and assembled from top to bottom by descending order of opening mesh. A fixed weight amount, for example 150 g, of the fraction to be analyzed is weighed with an analytical balance and placed on top of the widest sieve. By vibrating the sieving machine, the sample is conveyed through the various sieves. The sieving operation is run for a fixed amount of time, for example 15 minutes. The residues on each of the sieves are weighed with an analytical balance and related mathematically to the initial weight of materials. Notably, the d_{10}, d_{50} and d_{90} values can be calculated from the residues weights.

Preferably, the first fraction has a first d_{50} particle size value between 0.6 – 8.0 mm, more preferably between more preferably between 0.6 – 6.0 mm, more preferably between 0.8 – 6.0 mm, even more preferably between 0.9 - 4.0 mm, even more preferably between 1.0 – 3.0 mm, even more preferably between 1.0 – 2.0 mm.

Furthermore, the inventors have surprisingly found that by using a first fraction consisting of the first particles which have a specific particle size distribution, the composition (C) demonstrates excellent sintering properties.

This being said, the particle size distribution of this first fraction, as detailed above, can be further characterized by a first d_{90} and a first d_{10} particle size value.

Without being bound to this theory, the inventors believe that the particle size distribution of the first fraction, allows said first fraction to improve the packing of the particles, thereby improving the overall porosity of said first fraction.

Advantageously, the first fraction, as detailed above, has a first d_{10} particle size value equal to or higher than 100.0 µm, a first d_{50} particle size value of between 0.6 – 8.0 mm, and a first d_{90} particle size value equal to or less than 10.0 mm, preferably a first d_{10} particle size value equal to or higher than 200.0 µm a first d_{50} particle size value of between 0.8-6.0 mm, and a first d_{90} particle size value equal to or less than 8.0 mm, more preferably a first d_{10} particle size value equal to or higher than 300.0 µm a first d_{50} particle size value of between 1.0-2.0 mm, and a first d_{90} particle size value equal to or less than 4.0 mm.

According to one embodiment of the composition (C) according to the present invention, the particle size distribution of the first fraction, as detailed above, is characterized by a span value (SV) from 1.0 to 14.0, preferably from 1.0 to 8.0, more preferably from 1.0 to 6.0, even more preferably from 1.0 to 4.0.

Within the context of the present invention, the span value (SV) is defined as follows:

$$SV = \frac{(d_{90} - d_{10})}{d_{50}}$$

The span value (SV) characterizes the breadth of a particle size distribution. A small SV means that the particle size distribution is narrow, while a large SV means that the particle size distribution is broad.

As said above, the first fraction of the particulate mixture has a first chemical composition comprising, relative to the total weight of the first chemical composition:

- from 20.0 wt.% to 45.0 wt.% of at least one first magnesium oxide compound, as expressed in wt.% of MgO;
- from 40.0 wt.% to 60.0 wt.% of at least one calcium oxide compound, as expressed in wt.% of CaO; and
- from 5.0 wt.% to 12.0 wt.% of at least one iron oxide compound, as expressed in wt.% of Fe_2O_3.

Within the context of the present invention, the expression “at least one first magnesium oxide compound”, “at least one second magnesium oxide compound”, “at least one calcium oxide compound”, and “at least one iron oxide compound” are intended to denote one or more than one first magnesium oxide compound, one or more than one second magnesium oxide compound, one or more than one calcium oxide compound, and one or more than one iron oxide compound, respectively.

In the rest of the text, the expressions “first magnesium oxide compound”, “second magnesium oxide compound”, “calcium oxide compound”, and “iron oxide compound” are intended to denote one or more than one first magnesium oxide compound, one or more than one second magnesium oxide compound, one or more than one calcium oxide compound and one or more than one iron oxide compound, respectively.

Within the context of the present invention, the first magnesium oxide compound is understood to denote a first magnesium oxide containing compound, with the proviso that the amount of magnesium hydroxide in said first magnesium oxide containing compound is less than 1.0 wt.%, relative to the total weight of the first magnesium oxide.

Within the context of the present invention, the first magnesium oxide compound may also exist in different mineral phases.

Non-limiting example of the first magnesium oxide compound, mention may be made of magnesium oxide (MgO) and dolime.

Non-limiting example of suitable magnesium oxide based mineral phases that may be used mention may be made of periclase, magnesiowustite, and magnesioferrite.

Within the context of the present invention, the first calcium oxide compound is understood to denote a first calcium oxide containing compound.

Within the context of the present invention, the calcium oxide compound may also exist in different mineral phases.

Non-limiting example of calcium oxide compound, mention may be made of calcium oxide (CaO) and dolime.

Non-limiting example of suitable calcium oxide based mineral phase mention may be made of dicalcium silicate (C2S), tricalcium silicate (C3S), gehlenite and calcium-alumino-ferrite.

Within the context of the present invention, the iron oxide compound may also exist in different mineral phases.

Non-limiting example of iron oxide compound, mention may be made of iron oxide hematite (Fe_2O_3), wurstite (FeO) and magnetite (Fe_3O_4).

Non-limiting example of suitable iron oxide based mineral phase mention may be made of calcium ferrite, calcium-alumino-ferrite, magnesiowustite, and magnesioferrite.

In general, the amount of said magnesium oxide, calcium oxide and iron oxide compounds may be measured by X-Ray Fluorescence (XRF) chemical analysis, the results being expressed as oxides.

Advantageously, the amount of the first magnesium oxide in the first chemical composition, as detailed above, relative to the total weight of the first chemical composition, is equal to or more than 22.0 wt. %, more preferably equal to or more than 25.0 wt.%, even more preferably equal to or more than 30.0 wt.%, as expressed in wt.% of MgO.

Advantageously, the amount of the calcium oxide compound in the first chemical composition, as detailed above, relative to the total weight of the first chemical composition, is equal to or more than 42.0 wt. %, more preferably equal to or more than 45.0 wt.%, even more preferably equal to or more than 47.0 wt.%, as expressed in wt.% of CaO.

It is further understood that the upper limit of the amount of calcium oxide compound in the first chemical composition, as detailed above, is equal to or less than 57.0 wt.%, preferably equal to or less than 56.0 wt.%, more preferably equal to or less than 55.0 wt.%, as expressed in wt.% of CaO.

According to a preferred embodiment of the present invention, the amount of the calcium oxide compound in the first chemical composition, as detailed above, relative to the total weight of the first chemical composition, is from 42.0 to 57.0 wt.%, preferably from 45.0 to 56.0 wt.%, more preferably from 47.0 to 55.0 wt.%, as expressed in wt.% of CaO.

Advantageously, the amount of the iron oxide compound in the first chemical composition, as detailed above, relative to the total weight of the first chemical composition, is equal to or more than 5.5 wt. %, more preferably equal to or more than 6.0 wt.%, even more preferably equal to or more than 6.5 wt.%, as expressed in wt.% of Fe_2O_3.

It is further understood that the upper limit of the amount of iron oxide compound in the first chemical composition, as detailed above, is equal to or less than 11.5 wt.%, preferably equal to or less than 11.0 wt.%, more preferably equal to or less than 10.5 wt.%, as expressed in wt.% of Fe_2O_3.

According to a preferred embodiment of the present invention, the amount of the iron oxide compound in the first chemical composition, as detailed above, relative to the total weight of the first chemical composition, is from 5.5 to 11.5 wt.%, preferably from 6.0 to 11.0 wt.%, more preferably from 6.5 to 10.5 wt.%, as expressed in wt.% of Fe_2O_3.

According to one embodiment of the composition (C) of the present invention, the first chemical composition, relative to the total weight of the first chemical composition comprises:

- from 22.0 wt.% to 45.0 wt.% of at least one first magnesium oxide compound, as expressed in wt.% of MgO;
- from 42.0 wt.% to 57.0 wt.% of the at least one calcium oxide compound, as expressed in wt.% of CaO; and
- from 5.5 wt.% to 11.5 wt.%, of at least one iron oxide compound, as expressed in wt.% of Fe_2O_3;

preferably:

- from 25.0 wt.% to 45.0 wt.% of at least one first magnesium oxide compound, as expressed in wt.% of MgO;
- from 45.0 wt.% to 56.0 wt.% of the at least one calcium oxide compound, as expressed in wt.% of CaO; and
- from 6.0 wt.% to 11.0 wt.%, of at least one iron oxide compound, as expressed in wt.% of Fe_2O_3;

more preferably:

- from 30.0 wt.% to 45.0 wt.% of at least one first magnesium oxide compound, as expressed in wt.% of MgO;
- from 47.0 wt.% to 55.0 wt.% of the at least one calcium oxide compound, as expressed in wt.% of CaO; and
- from 6.5 wt.% to 10.5 wt.%, of at least one iron oxide compound, as expressed in wt.% of Fe_2O_3.

The first fraction of the particulate mixture comprised in the composition (C) of the present invention may be commercially available or may be synthetically prepared. Said preparation of the first fraction may be carried out using conventional method known to the skilled in the art such as by providing a feed material comprising dead-burnt dolomite and an iron oxide compound, thereby providing a particulate mixture which is then sintered. Example of such conventional method may be notably found in GB 2519702A, the whole content of which is herein incorporated by reference.

The first fraction of the particulate mixture comprised in the composition (C) of the present invention may be synthetically prepared by providing a feed material consisting of dead-burnt dolomite and an iron oxide-containing compound, such as for example mill scale. The feed material hereby obtained may undergo a sintering step. The sintered feed material thereby obtained may undergo a crushing and/or a screening and/or a grading process to obtain the first fraction of the particulate mixture comprised in the composition (C), as detailed above.

As said above, the second fraction of the particulate mixture comprised in the composition (C) has a second d_{50} particle size, wherein the ratio between the second d_{50} particle size value of the second fraction and the first d_{50} particle size value of the first fraction is from 0.05 to 0.95.

The inventors have surprisingly found that the second d_{50} particle size value of the second fraction consisting the particulate mixture comprised in the composition (C) allows said composition (C) to show a low slag penetration within said composition (C), and to consequently show an excellent resistance to slag corrosion, as demonstrated in the working examples. Furthermore, the inventors have found that the second d_{50} particle size value of the second fraction consisting the particulate mixture comprised in the composition (C) has an influence on the shrinkage of the composition (C), when the composition (C) is sintered and used as hot-repair materials.

Thus, the second fraction of the particulate mixture comprised in the composition (C) advantageously has a second d_{50} particle size value, wherein the ratio between the second d_{50} particle size value of the second fraction and the first d_{50} particle size value of the first fraction is of at least 0.05, or at least 0.08, or at least 0.95, or at least 0.10.

It is further understood that the second fraction of the particulate mixture comprised in the composition (C) advantageously has a second d_{50} particle size value, wherein the ratio between the second d_{50} particle size value of the second fraction and the first d_{50} particle size value of the first fraction is of at most 0.95, or at most 0.85, or at most 0.75, or at most 0.65, or at most 0.50 or at most 0.45, or at most 0.40 or at most 0.30.

Thus, the second fraction of the particulate mixture comprised in the composition (C) advantageously has a second d_{50} particle size value, wherein the ratio between the second d_{50} particle size value of the second fraction and the first d_{50} particle size value of the first fraction is from 0.08 to 0.85, or from 0.10 to 0.75, or from 0.10 to 0.65, or from 0.10 to 0.50, or from 0.10 to 0.45, or from 0.10 to 0.40, or from 0.10 to 0.30.

The inventors have also surprisingly found that it is this specific ratio between the second d_{50} particle size value of the second fraction and the first d_{50} particle size value of the first fraction, as detailed above, that enables the composition (C) of the present invention to have a good flowability (i.e. a good cohesive index, a good Hausner ratio, and a good difference of density between the tapped density and the bulk density). A good flowability of the composition (C) further enables the composition (C) of the present invention to be particularly processable. In other words, the inventors have surprisingly found that this specific ratio between the second d_{50} particle size value of the second fraction and the first d_{50} particle size value of the first fraction, as detailed above, reduces/prevents the agglomeration of the particles within the composition (C) upon use, storage or transport of the composition (C), as demonstrated in the working examples.

In an embodiment of the present invention, the particle size distribution of the second fraction, as detailed above, is characterized by a span value (SV) of at most 10.0, or of at most 7.5, or of at most 6.0.

It is further understood that the particle size distribution of said second fraction is advantageously characterized by a span value (SV) of at least 1.0, or at least 1.1, or of at least 1.2.

In a preferred embodiment of the composition (C) according to the present invention, the particle size distribution of the second fraction, as detailed above, is characterized by a span value (SV) from 1.0 to 10.0, or from 1.1 to 7.5, or from 1.2 to 6.0.

Advantageously, the second fraction, as detailed above, has a d_{50} particle size value of at least 50.0 µm, or of at least 55.0 µm, or of at least 60.0 µm, or of at least 70.0 µm.

It is further understood that the d_{50} particle size value of the second fraction is advantageously of at most 1000.0 µm, or at most 750.0 µm, or of at most 600.0 µm.

In a preferred embodiment of the composition (C) according to the present invention, the second fraction, as detailed above, has a d_{50} particle size value of between 50.0 µm and 1000.0 µm, or of between 60.0 µm and 750.0 µm, or of between 70.0 µm and 600.0 µm.

Advantageously, the second fraction, as detailed above, has a d_{10} particle size value of at least 10.0 µm, or of at least 13.0 µm, or of at least 15.0 µm.

It is further understood that the d_{10} particle size value of the second fraction is advantageously of at most 50.0 µm, or at most 40.0 µm, or of at most 30.0 µm.

In a preferred embodiment of the composition (C) according to the present invention, the second fraction, as detailed above, has a d_{10} particle size value of between 10.0 µm and 50.0 µm, or of between 13.0 µm and 40.0 µm, or of between 15.0 µm and 30.0 µm.

Advantageously, the second fraction, as detailed above, has a d_{90} particle size value of at least 500.0 µm, or of at least 600.0 µm, or of at least 700.0 µm.

It is further understood that the d_{90} particle size value of the second fraction is advantageously of at most 1500.0 µm, or at most 1250.0 µm, or of at most 1100.0 µm.

In a preferred embodiment of the composition (C) according to the present invention, the second fraction, as detailed above, has a d_{90} particle size value of between 500.0 µm and 1500.0 µm, or of between 600.0 µm and 1250.0 µm, or of between 700.0 µm and 1100.0 µm.

As said above, the total content of the first and the second magnesium oxide compound in the particulate mixture, relative to the total weight of the particulate mixture, is from 30.0 to 80.0 wt.%, as expressed in wt.% of MgO.

Advantageously, the total content of the first and the second magnesium oxide compounds in the particulate mixture, relative to the total weight of the particulate mixture, is equal to or higher than 32.0 wt.%, more preferably equal to or higher than 34.0 wt.%, even more preferably equal to or higher than 37.0 wt.%, as expressed in wt.% of MgO.

It is further understood that the upper limit the total content of magnesium oxide compounds in the particulate mixture comprised in the composition (C), relative to the total weight of the particulate mixture, is equal to or less than 75.0 wt.%, preferably equal to or less than 70.0 wt.%, more preferably equal to or less than 65.0 wt.%, even more preferably equal to or less than 60.0 wt.%, even more preferably equal to or less than 55.0 wt.%, even more preferably equal to or less than 52.0 wt.%, even more preferably equal to or less than 50.0 wt.%, as expressed in wt.% of MgO.

According to a preferred embodiment of the composition (C) of the present invention, the total content of the first and the second magnesium oxide compounds, relative to the total weight of the particulate mixture, is from 32.0 to 75.0 wt.%, preferably from 34.0 to 70.0 wt.%, more preferably from 34.0 to 65.0 wt.%, more preferably from 37.0 to 65.0 wt.%, even more preferably from 37.0 to 60.0 wt.%, even more preferably from 37.0 to 55.0 wt.%, even more preferably from 37.0 to 50.0 wt.%, even more preferably from 37.0 to 48.0 wt.%, as expressed in wt.% of MgO.

It goes without saying that the content of the second magnesium oxide compound in the particulate mixture of the composition (C) of the present invention, is determined by the content of the first magnesium oxide in the first chemical composition and the total content of all the magnesium oxide compounds present in the composition (C).

As detailed above, the second chemical composition of the second particles comprises at least one second magnesium oxide compound.

According to certain embodiments of the composition (C) of the present invention, the amount of the second magnesium oxide compound in the second chemical composition, as detailed above, relative to the total weight of the second chemical composition, is equal to or more than 20.0 wt.%, more preferably equal to or more than 30.0 wt.%, even more preferably equal to or more than 40.0 wt.%, even more preferably equal to or higher than 60.0 wt.%, as expressed in wt.% of MgO .

It is further understood that the upper limit of the amount of the second magnesium oxide compound in the second chemical composition, as detailed above, relative to the total weight of the second chemical composition is equal to or less than

80.0 wt.%, preferably equal to or less than 75.0 wt.%, more preferably equal to or less than 70.0 wt.%, as expressed in wt.% of MgO.

According to preferred embodiment of the composition (C) of the present invention, the amount of the second magnesium oxide compound in the second chemical composition, as detailed above, relative to the total weight of the second chemical composition is from 20.0 to 80.0 wt.%, preferably from 30.0 to 75.0 wt.%, more preferably from 40.0 to 70.0 wt.%, even more preferably from 60.0 to 70.0 wt.%, as expressed in wt.% of MgO.

The second magnesium oxide compound may be identical to or different from the first magnesium oxide compound, as detailed above.

Within the context of the present invention, the second magnesium oxide compound is understood to denote a second magnesium oxide containing compound, with the proviso that the amount of magnesium hydroxide in said second magnesium oxide containing compound is less than 1.0 wt.%, relative to the total weight of the second magnesium oxide.

Within the context of the present invention, the second magnesium oxide compound may also exist in different mineral phases.

Non-limiting example of the second magnesium oxide compound, mention may be made of magnesium oxide (MgO), and dolime.

Non-limiting example of suitable second magnesium oxide based mineral phases that may be used mention may be made of periclase, magnesiowustite, and magnesioferrite.

According to a preferred embodiment of the composition (C) of the present invention, the second chemical composition further comprises at least one second calcium compound which may be identical or different to the calcium oxide compound of the first fraction.

Within the context of the present invention, the expression, “at least one second calcium oxide compound”, is intended to denote one or more than one second calcium oxide compound.

In the rest of the text, the expression “second calcium oxide compound”, is intended to denote one or more than one second calcium oxide compound.

Within the context of the present invention, the second calcium oxide compound is understood to denote a second calcium oxide containing compound.

Within the context of the present invention, the second calcium oxide compound may also exist in different mineral phases.

Non-limiting example of calcium oxide compound, mention may be made of calcium oxide (CaO) and dolime.

Non-limiting example of suitable calcium oxide based mineral phase mention may be made of dicalcium silicate (C2S), dicalcium trisilicate (C3S) and calcium-alumino-ferrite.

Advantageously, the amount of the calcium oxide compound in the second chemical composition, as detailed above, relative to the total weight of the second chemical composition, is equal to or more than 42.0 wt.%, more preferably equal to or more than 45.0 wt.%, even more preferably equal to or more than 47.0 wt.%, as expressed in wt.% of CaO.

It is further understood that the upper limit of the amount of calcium oxide compound in the second chemical composition, as detailed above, relative to the total weight of the second chemical composition, is equal to or less than 57.0 wt.%, preferably equal to or less than 56.0 wt.%, more preferably equal to or less than 55.0 wt.%, as expressed in wt.% of CaO.

According to a preferred embodiment of the present invention, the amount of the calcium oxide compound in the second chemical composition, as detailed above, relative to the total weight of the second chemical composition, is from 42.0 to 57.0 wt.%, preferably from 45.0 to 56.0 wt.%, more preferably from 47.0 to 55.0 wt.%, as expressed in wt.% of CaO.

The second fraction of the particulate mixture comprised in the composition (C) of the present invention may be commercially available, may be synthetically prepared from raw materials, such as dolime, or may originate from recycled materials such as recycled refractory bricks from iron and/or steel processes or recycled dust originating from mineral processes such as magnesia burning processes, or a mixture thereof.

Non-limiting examples of recycled refractory bricks from iron and/or steel processes mention may be made of recycled basic refractory bricks, such as recycled magnesia and doloma (also called “dolime”) refractory bricks, said recycled basic refractory bricks may originate from the dismantling of used refractory linings of vessels used in the iron and steel-making industry, said vessel may be an Electric Arc

Furnace (EAF) vessel, a Basic Oxygen Furnace (BOF) vessel, an Argon Oxygen Decarburization (AOD) vessel, a transfer ladle, a casting ladle or a tundish.

Preferably, the second fraction of the particulate mixture comprised in the composition (C) of the present invention is synthetically prepared from raw materials, such as dolime, or originates from recycled materials such as magnesia and doloma recycled refractory bricks from iron and/or steel processes, recycled dust originating from mineral processes such as magnesia burning processes, or a mixture thereof.

The second fraction originating from recycled materials, allows the composition (C) to require less virgin material. The composition (C), when said second fraction originates from recycled materials is therefore part of a circular economy. Not only the second fraction originating from recycled materials replaces virgin material, they also include another dolomite quality, which allows in higher utilization of purer form of dolomite. Consequently, the quarry life is improved. Finally, said second fraction originating from recycled materials allows to reduce the overall CO_2 emission, which is an inevitable by-product of the manufacturing of virgin material. The amount of hazardous wastes being predominantly landfilled is also reduced by valorizing said wastes.

Most preferably, the second fraction of the particulate mixture comprised in the composition (C) of the present invention originates from recycled materials selected from the group consisting of recycled magnesia and doloma refractory bricks.

Even more preferably, the second fraction of the particulate mixture comprised in the composition (C) of the present invention originates from doloma refractory bricks.

Even more preferably, the second fraction of the particulate mixture comprised in the composition (C) of the present invention originates from recycled materials selected from the group consisting of recycled doloma refractory bricks from an Argon Oxygen Decarburization (AOD) vessel.

When the second fraction of the particulate mixture comprised in the composition (C) of the present invention originates from recycled materials, as defined above, any using conventional method known to the skilled in the art, such as milling techniques, grinding techniques, sieving techniques, grading techniques, or a combination thereof, may be used to obtain said second fraction.

According to one embodiment of the composition (C) of the present invention, the first fraction and the second fraction, as detailed above, are present in a weight ratio first fraction/second fraction of between 90/10 to 50/50, preferably of between 85/15 to 55/45, more preferably of between 75/25 to 60/40.

The inventors have also found that total content of aluminum oxide compound and silicon oxide compound in the particulate mixture comprised in the composition (C) should be kept low, as it is inferred to lead to increase the slag penetration inside the composition (C), when such composition is used as hot-repair refractory material and when such hot-repaired refractory materials are used in a subsequent iron and steel making process, as demonstrated in the working examples.

According to a preferred embodiment of the composition (C) of the present invention, the particulate mixture is substantially free of aluminum oxide compound and silicon oxide compound.

Within the context of the present invention, the expression "substantially free of aluminum oxide compound and silicon oxide compound is intended to denote that the total content of aluminum oxide compound and silicon oxide compound, relative to the total weight of the particulate mixture, is equal to or less than 5.5 wt.%, preferably equal to or less than 5.0 wt.%, more preferably equal to or less than 4.0%, even more preferably equal to or less than 3.0 wt.%, as expressed in wt.% of Al_2O_3.and SiO_2.

Within the context of the present invention, the aluminum oxide compound may also exist in different mineral phases.

Non-limiting example of the aluminum oxide compound, mention may be made of aluminum oxide (Al_2O_3).

Non-limiting example of suitable aluminum oxide based mineral phases that may be used mention may be made of calcium- aluminates such as tricalcium aluminate, gehlenite, and the like.

Within the context of the present invention, the silicon oxide compound may also exist in different mineral phases.

Non-limiting example of the silicon oxide compound, mention may be made of silicon oxide (SiO_2).

Non-limiting example of suitable silicon oxide based mineral phases that may be used mention may be made of calcium silicates such as dicalcium silicate (C2S), tricalcium silicate (C3S), gehlenite, and the like.

In general, the amount of said aluminum oxide and silicon oxide based compounds may be measured by X-Ray Fluorescence (XRF) chemical analysis, the results being expressed as oxides.

According to certain embodiments of the present invention, the composition (C), comprises a particulate mixture consisting of at least one first fraction consisting of first particles having a first chemical composition and at least one second fraction consisting of second particles having a second chemical composition, wherein the first chemical composition is different from the second chemical composition.

According to a preferred embodiment of the present invention, the composition (C), comprises a particulate mixture consisting of at least one first fraction consisting of first particles having a first chemical composition and at least one second fraction consisting of second particles having a second chemical composition, wherein the first fraction has a first d_{50} particle size value of between 1.0 – 3.0 mm and the at least one second fraction has a second d_{50} particle size value of between 70.0 µm and 600.0 µm and wherein the ratio between the second d_{50} particle size value of the second fraction and the first d_{50} particle size value of the first fraction is from 0.10 to 0.30, and

wherein the first chemical composition, relative to the total weight of the first chemical composition comprises:

- from 20.0 wt.% to 45.0 wt.% of at least one first magnesium oxide compound, as expressed in wt.% of MgO;
- from 40.0 wt.% to 60.0 wt.% of at least one calcium oxide compound, as expressed in wt.% of CaO;
- from 5.0 wt.% to 12.0 wt.% of at least one iron oxide compound, as expressed in wt.% of Fe_2O_3, and

wherein the second chemical composition comprises at least one second magnesium oxide compound, wherein the total content of the first and the second magnesium oxide compounds in the particulate mixture, relative to the total weight of the particulate mixture is from 30.0 to 48.0 wt.%, as expressed in wt.% of MgO.

According to a preferred embodiment of the present invention, the composition (C) suitable for hot repairing refractories, consists essentially of the particulate mixture, wherein the particulate mixture consist of the at least one first fraction consisting of the first particles having the first chemical composition, as detailed above and at the least one second fraction consisting of the second particles having the second chemical composition, as defined above, wherein the first fraction has a first d_{50} particle size value between 1.0 – 3.0 mm and the at least one second fraction has a second d_{50} particle size value of between 70.0 µm and 600.0 µm and, and wherein the ratio between the second d_{50} particle size value of the second fraction and the first d_{50} particle size value of the first fraction is from 0.05 to 0.95, and

wherein the first chemical composition, relative to the total weight of the first chemical composition comprises:

- from 20.0 wt.% to 40.0 wt.% of at least one first magnesium oxide compound, as expressed in wt.% of MgO;
- from 40 wt.% to 60 wt.% of at least one calcium oxide compound, as expressed in wt.% of CaO;
- from 5.0 wt.% to 12.0 wt.% of at least one iron oxide compound, as expressed in wt.% of Fe_2O_3, and

wherein the second chemical composition comprises at least one second magnesium oxide compound, wherein the total content of the first and the second magnesium oxide compounds in the particulate mixture, relative to the total weight of the particulate mixture is from 30.0 to 48.0 wt.%, as expressed in wt.% of MgO.

For the purpose of the present invention, the expression “consist essentially of” is intended to denote that any additional ingredient different from the particulate mixture, as defined above is present in minor amounts in said composition (C), being understood that the additional ingredients do not substantially modify the properties of said composition (C).

According to other embodiments of the present invention, the composition (C) may further comprise at least one oiling compound.

Within the context of the present invention, the expressions “at least one oiling compound” is intended to denote one or more than one oiling compound. Said oiling compounds hinder and even prevent the hydration of particulate mixture. Furthermore, said oiling compound, when used in the composition (C) suitable for hot-repairing refractories, helps to increase the angle of repose of the particulate mixture, as defined above, and improves the cohesion of the particles in the particulate mixture, as defined above, when the composition (C), as detailed above, is applied to a refractory lining in a vessel, thereby enhancing the process.

In the rest of the text, the expression “oiling compound” is understood, for the purpose of the present invention, both in the plural and in the singular form, that is to say that the composition (C) of the present invention further comprises one oiling compound or more than one oiling compound.

Said oiling compounds are known to those skilled in the art of hot-repair materials. Non-limiting examples of suitable oiling compound, mention may be made of mineral oil; petroleum-based oil such as heavy fuel oil, gas or gasoline; vegetable based oil such as flaxseed oil; and petroleum jelly such as Vaseline®.

Preferably, the oiling compound in the composition (C) according to the present invention is selected from mineral oil and petroleum jelly.

As to the amounts of the oiling compounds, it is further understood that the skilled person in the art will practice said oiling compounds in a suitable amount according to standard and general practice known by said skilled person in the art.

In general, the amount of the oiling compound, as detailed above, when present, is from 0.50 to 4.00 wt.%, or from 0.75 to 3.00 wt.%, or from 0.90 to 2.00 wt.%, relative to the total weight of the composition (C).

According to certain embodiments of the present invention, the composition (C) suitable for hot repairing refractories, comprises or consists essentially of, relative to the total weight of the composition (C), from 96.0 wt.% to 99.5 wt.% of the particulate mixture, as detailed above, and from 0.5 wt.% to 4 wt.% of the oiling compound, as detailed above.

For the purpose of the present invention, the expression “consist essentially of” is intended to denote that any additional ingredient different from the particulate mixture, as defined above, and the oiling compound, as detailed above, is present in minor amounts in said composition (C), being understood that the additional ingredients do not substantially modify the properties of said composition (C).

Another aspect is a composition (C) suitable for hot repairing refractories, comprising a particulate mixture consisting of at least one first fraction consisting of first particles and at least one second fraction consisting of second particles, wherein the first fraction has a first d_{50} particle size value between 0.5 – 10 mm and the second fraction has a second d_{50} particle size value, wherein the ratio between the second d_{50} particle size value of the second fraction and the first d_{50} particle size value of the first fraction is from 0.05 to 0.95, and wherein said first fraction has the second chemical composition as described herein-above,

and wherein said second fraction has the first chemical composition as described herein-above.

All definitions and preferences, as described above, equally apply here in this aspect of the present invention.

The methods for the manufacturing of the composition (C) are also an aspect of the present invention.

It is further understood that all definitions and preferences, as described above, equally apply for all further embodiments, as described below.

The composition (C) of the present invention can be prepared by a variety of methods known in the art. For manufacturing the composition (C) of the present invention, several methods known in the art may be adequately used.

In one embodiment of the present invention, the method for the manufacturing of the composition (C), as detailed above, comprises at least a step of intimate admixing of the at least one first fraction, as detailed above, the at least one second fraction, as detailed above, and optionally the at least one oiling compound, as detailed above.

The inventors have found that such method for the manufacturing of the composition (C) allows an easy and energy efficient tailoring of the chemical composition and particle size distribution of said composition (C). Consequently, the composition (C) can be easily adapted in term of for example chemical composition, to the refractory lining onto which the composition (C) is applied to.

In a preferred embodiment of the present invention, the method for the manufacturing of the composition (C), as detailed above, comprises the step of intimate admixing:

- from 48.0 to 87.0 wt.% of at least one first fraction, as detailed above;
- from 9.0 to 48.0 wt.% of at least one second fraction, as detailed above; and
- optionally at least one oiling compound, as detailed above;

wherein all wt.% are relative to the total weight of the composition (C).

In a more preferred embodiment of the present invention, the method for the manufacturing of the composition (C), as detailed above, comprises the step of intimate admixing:

- from 58.0 to 72.0 wt.% of at least one first fraction, as detailed above;
- from 24.0 to 38.0 wt.% of at least one second fraction, as detailed above; and
- optionally at least one oiling compound, as detailed above,

wherein all wt.% are relative to the total weight of the composition (C).

Typically said intimate admixing, as detailed above, may be carried out by using traditional mixers and blenders, high intensity mixers, electric stirrers and pan mill.

It is understood that the skilled person in the art will carry out said intimate admixing according to general practice, such as notably using optimal times, speeds, weights, volumes and batch quantities.

Furthermore, it is understood that any order of intimate admixing of the various components as comprised in the composition (C), as detailed above, is acceptable.

If desired, the at least one first fraction, as detailed above, can first be mixed with the at least one second fraction, as detailed above, thereby forming the particulate mixture as defined above, said particulate mixture is then further optionally mixed with the at least one oiling compound, as defined above.

If desired, the at least one first fraction, as detailed above, can first be optionally mixed in at least part of the at least one oiling compound, thereby forming a first premix, said first premix is then further mixed with the at least one second fraction, as detailed above, and optionally the remaining part of the at least one oiling compound.

If desired, the at least one second fraction, as detailed above, can first be optionally mixed in at least part of the at least one oiling compound, thereby forming a second premix, said second premix is then further mixed with the at least one first fraction, as detailed above, and optionally the remaining part of the at least one oiling compound.

According to a preferred embodiment of the method for the manufacturing of the composition (C) of the present invention, the at least one first fraction, as detailed above, is first mixed with the at least one second fraction, as detailed above, thereby forming the particulate mixture as defined above, said particulate mixture is then further optionally mixed with the at least one oiling compound, as defined above.

Another aspect of the present invention is a method for hot-repairing a refractory lining [refractory lining (L) herein-after], wherein said refractory lining is hot-repaired with the composition (C), as detailed above, and wherein the composition (C) is applied to the refractory lining (L).

It is further understood that all definitions and preferences, as described above, equally apply for all further embodiments, as described below.

In general, lining the inner wall of a vessel used in an iron or steel-making process with a refractory lining is crucial to preclude the direct contact of the vessel with the molten metals or slag. The refractory lining consequently allows theinner wall of the vessel to be protected from the high temperature and corrosive environment of the molten metal and slags inside the vessel.

Said refractory linings are known to those skilled in the art, and may notably include refractory lining originating from refractory bricks and/or refractory lining originating from monolithic refractories.

When a refractory lining originates from refractory bricks, said refractory bricks may be commercially available or may be prepared by using conventional methods known to the skilled in the art such as pressing and shaping particulate refractory materials in the form of bricks.

In general, the refractory lining originating from refractory bricks may be lined by assembling said refractory bricks onto the inner wall of a vessel.

When a refractory lining originates from monolithic refractories, said monolithic refractories may be commercially available or may be prepared by using conventional methods known to the skilled in the art and notably include the steps of mixing a particulate refractory material with optionally any binder, any filler and/or any additives known to the skilled in the art, to tailor the properties of the monolithic refractory.

In general, the refractory lining originating from monolithic refractories may be prepared from unshaped monolithic refractories in particulate form, using conventional methods known to the skilled in the art such as pouring, troweling, gunning, ramming, vibrating and injecting.

It is understood that the refractory lining may also be classified based on the interaction of the particulate refractory materials comprised in the refractory lining with water, that is to say acid, basic and neutral refractory linings.

Non-limiting examples of suitable refractory lining (L) mention may be made of acid refractory linings comprising alumina-silicate, silica or zircon based particulate refractory materials, neutral refractory lining comprising chromia or alumina-based particulate refractory materials, and basic refractory linings comprising magnesia, dolomite, magnesia-chrome or spinel-based particulate refractory materials.

Preferred refractory lining (L) according to the method of the present invention are basic refractory lining (L) comprising magnesia, magnesia-chrome, dolomite or spinel-based particulate refractory materials. Most preferred refractories lining (L) is a basic refractory lining comprising magnesia, magnesia-chrome or dolomite-based particulate refractory materials.

As said above, refractory lining are in general lined onto the inner wall of a vessel used in an iron or steel-making process.

Non-limiting examples of suitable vessel mention may be made of an Electric Arc Furnace (AOD) vessel, a Basic Oxygen Furnace (BOF) vessel, an Argon Oxygen Decarburization (AOD) vessel, a transfer ladle, a casting ladle or a tundish.

Preferably, the refractory lining (L) of the method of the present invention is lined onto an EAF vessel.

Among the suitable manners for applying the composition (C), as detailed above, to the refractory lining (L), as detailed above, mention can be notably made of conventional application method known to the skilled in the art of iron and steel-making industry such as fettling, spraying, or gunning the composition (C), by using conventional equipment such as a dedicated fettling machines.

In general, a fettling machine comprises a hopper, said hopper is loaded with the composition (C). The fettling machine thereby applies the composition to the refractory lining (L).

The inventors have surprisingly found that when the composition (C) according to the present invention is applied to the refractory lining (L) by using a fettling machine, the hopper dispenses the composition without clogging. Consequently, the inventors have found that the fettling machine can apply the composition (C) to the refractory lining (L) in an homogeneous and evenly way.

Without being bound to this theory, it is inferred that the specific ratio between the second d_{50} particle size value of the second fraction and the first d_{50} particle size value of the first fraction of the particulate mixture comprised in the composition (C), as detailed above, enables an optimal packing of said composition (C) when loaded in the hopper comprised in the fettling machine. In other words, the composition (C) according to the present invention, when applied to the refractory (L) shows good processability and flowability during the method for hot-repairing a refractory lining according to the present invention.

It is understood that the skilled person in the art will carry out said application of the composition (C), as detailed above, according to general practice, such as notably using optimal times, weights, volumes and batch quantities.

It is further understood that when the composition (C) is applied by using conventional equipment such as dedicated fettling machine, the skilled person in the art will carry out said application of the composition (C), as detailed above, according to general practice, such as notably the flow rate, the angle to apply said composition (C), and the quantity of the composition (C) to apply.

In general, in a method for hot-repairing refractory linings, hot repair materials (also called fettling materials), are applied onto hot refractory linings.

Advantageously, the composition (C) of the method for hot-repairing a refractory lining (L) according to the present invention, is applied on hot refractory lining (L) having a temperature of at least 1200 °C, or at least 1300 °C, or at least 1400 °C.

Preferably, the composition (C) is applied, as detailed above, on hot refractory lining (L) which have been previously used as refractory lining in a vessel in an steel making process, and wherein the vessel have been previously tapped of its molten iron or/steel and its slag.

This allows the refractory lining to have the adequate temperature, without having to heat the refractory lining, with is without doubt time consuming and requires high energy.

The inventors have surprisingly found that the composition (C), as detailed above, when applied to the hot refractory lining (L), as defined above, shows excellent sintering properties, as well as good shrinkage and corrosion resistance, which allows the sintered composition (C) to patch the eroded refractory lining (L), thereby prolonging the life of said refractory lining (L) before dismantling.

Another aspect of the present invention is a sintered hot repair-material obtained by the method for hot-repairing a refractory lining (L), as detailed above, wherein said refractory lining (L) is treated with the composition (C), as detailed above.

It is further understood that all definitions and preferences, as described above, equally apply for all further embodiments, as described below.

Another aspect of the present invention is the use of the composition (C), as detailed above, in the method for hot-repairing the refractory lining (L), as detailed above.

It is further understood that all definitions and preferences, as described above, equally apply for all further embodiments, as described below.

EXAMPLES

The invention will be now described in more details, whose purpose is merely illustrative and are not intended to limit the scope of the invention.

Particle size measurements – general procedure

The particle size of each of the fractions disclosed-below were measured by mechanical sieving measurements according to the general procedure described herein-after.

Mechanical sieving measurement was based on the mechanical separation of a fraction on a series of superimposed sieves and was determined with an automated mechanical sieving device. The automatic sieving device comprised means for mounting a nest of sieves, which was located on a vibrating sieve shaker. The sieves are superimposed and assembled from top to bottom by descending order of opening mesh.

150.0 g of the fraction to be analyzed was weighed with an analytical balance and placed on top of the widest sieve. By vibrating the sieving machine, the sample was conveyed through the various sieves. The sieving operation was run for 15 minutes and the residues on each of the sieves were weighted with an analytical balance and related mathematically to the initial weight of materials.

Preparation of the fractions 1 and 2

First fractions 1A, 1B and 1C were prepared by sieving a sintered admixture of dead-burned dolomite and a Fe_2O_3 compound. Said first fraction 1A consisted of first particles having each a first chemical composition and particles size values as mentioned in Table 1. All contents are given in wt.%, unless stated otherwise. All chemical compounds are expressed as oxide equivalent, unless stated otherwise. All d_{xx} particle size values are given in µm, unless stated otherwise.

The second fractions were synthetically prepared from dolime or originates from recycled doloma refractory bricks.

In particular, fractions 2A and 2D originate from recycled doloma refractory bricks which were crushed, grinded, and sieved to obtain the desired particle size.

The fractions 2B, 2C and 2E originate from dust from dolime production processes.

(Said second fractions 2A, 2B, 2C, 2D and 2E consisted of second particles having each a second chemical composition and particles size values as mentioned in Table 1 and Table 2.

Second fraction 2CE originated from dust particles from dolime production processes. Said second fraction 2CE consisted of second particles having each a second chemical composition and particles size values as mentioned in Table 2.

All contents are given in wt.%, unless stated otherwise. All chemical compounds are expressed as oxide equivalent, unless stated otherwise.

Examples 1, 2, 4 and comparative examples 1-3

A. Preparation of the composition (C) of examples 1, 2, 4 and comparative examples 1-3

Compositions (C) according to examples 1, 2 and 4 (E1, E2, and E4) were prepared by mixing the first fraction 1A or 1B with the second fraction 2A and/or 2B and/or 2C in amounts as summarized in Table 1. All contents are given in wt.%, unless stated otherwise.

B. Properties of the composition (C) of examples 1, 2 and 4 (E1, E2, and E4)

Thermodynamic calculations

Thermodynamic calculations of the compositions (C) of examples 1, 2 and 4 and comparative examples 1-2 were performed using a simulation program. The purpose is to predict the interaction between a liquid slag and each of the compositions (C) of examples 1, 2 and 4 and comparative examples 1-2, by notably determining the phase fraction of liquid slag at a close range to the composition (C), which is virtually lined.

These thermodynamic calculations were carried out by using the Thermo-Calc Software 2020b and TCOX10 Metal Oxide Solutions Database. The interfacial reactions between each of the compositions (C) of Examples 1, 2 and 4 (E1, E2 and E4) and each of the comparative examples 1-2 (CE1 and CE2) and a liquid slag, at a temperature of 1550°C were simulated. The chemical composition of the liquid slag is summarized in the following Table A herein-below:

Table ASlag Compound	Chemical composition expressed in oxide equivalent (wt.%)
CaO	38.9
MgO	9.7
Al_2O_3	6.1
SiO_2	15.7
Fe_2O_3	26.0
Mn_3O_4	2.4
TiO_2	0.9
P_2O_5	0.3
Sum	100.0

The thermodynamic simulations are based on the assumptions that there is a thermodynamic equilibrium between each of the composition (C) of the examples 1, 2 and 4 (E1, E2, and E4) and each of comparative examples 1-2 (CE1-CE2) and the liquid slag, as well as a homogeneous chemistry inside the slag and the composition (C).

The penetration of the liquid slag into the composition (C) was investigated by measuring the slag liquid fraction at close range to the composition (C), which is virtually lined, *i.e.* at a value x=0.9, which corresponds to 90% of composition (C) and 10% liquid slag indicating the quantity of liquid fraction and allowing quantitative comparisons between different refractory compositions in relation to liquification as a result of reaction with said liquid slag. Compositions according to examples 1,2 and 4 (E1, E2 and E4) and are compared to comparative example 1 (CE1) and to comparative example 2 (CE2).

Additional properties (circular economy and quarry life)

The impact of examples 1, 2 and 4 and comparative examples 1-3 in view of their circular economy and quarry life were evaluated with reference to comparative example 1.

The performance assessment of each example 1, 2, and 4 and comparative example 1, 2 and 3 was done by counting the number of “+” and “–” signs obtained in each test, the “-” sign being counted as negative.

The circular economy evaluation is intended to consider the recycling and the reuse of waste materials, with the aim to reduce the use of virgin materials and thereby reducing the amount of waste materials and reducing the cost.

The quarry life evaluation is intended to relate to percentage of recycled materials used as well as the ability to specifically use less primary material mined from the quarry and to less landfilling of mining wastes back to the quarry. Furthermore, the quarry life evaluation is linked to the ability to reduce the overall CO_2 emission, which is an inevitable by-product of the manufacturing of virgin material from primary materials.

Table 1

	Composition (C)					
	CE1	**E1**	**E2**	**E4**	**CE2**	**CE3**
Particulate mixture						
Fraction 1A (wt.%)	100	75	60	75	0	75
%CaO =53.7% d_{10} = 400.0 µm % MgO = 37.0 % d_{50} = 1300.0 µm %Fe_2O_3 = 7.5% d_{90} = 3000.0 µm						
Fraction 1B (wt.%)	0	0	0	0	100	0
%CaO =51.2% d_{10} = 400.0 µm % MgO = 35.3% d_{50} = 1300.0 µm %Fe_2O_3 = 9.9% d_{90} = 3000.0 µm						
Fraction 2A (wt.%)	0	0	0	25	0	0
%CaO = 24.7 % d_{50} = 900 µm % MgO = 66.6 %						
Fraction 2B (wt.%)	0	0	0	0	0	25
%CaO = 29.8 % d_{10} = 3.0 µm % MgO = 20.9 % d_{50} = 20-30 µm d_{90} =100 µm						
Fraction 2C (wt.%)	0	25	40	0	0	0
%CaO = 42.9 % d_{10} = 10-20 µm % MgO = 30 % d_{50} = 100 µm d_{90} = 400-500 µm						
Chemical composition of the particulate mixture						
% (SiO_2) + % Al_2O_3	< 1.7	<1.6	<2.7	<2.3	<4	<4.4
% MgO	37	35.3	34.2	44.4	35.3	33.0
Thermodynamic calculations						
Liquid slag fraction at 0.9 refractory fraction	0.28	0.26	0.25	0.26	0.38	NA
Ratio d_{50} second fraction / d_{50} first fraction	-	0.08	0.08	0.69	-	0.015-0.023
Additional properties						
Circular economy	Ref	+	++	+	=	+
Quarry life	Ref	+	+	+	+	+

It was observed through thermodynamic simulations that compositions (C) according to examples 1, 2 and 4 all show similar or lower liquid slag fraction at 0.9 refractory fraction as compared to comparative example 1.

It should be noted that the liquid slag fraction is an indicator for the penetration of the liquid slag to the refractory. In other words, the lower the liquid slag fraction at 0.9 refractory fraction, the lower the penetration.

Hence it was demonstrated that the composition (C) according to examples E1, E2, and E4 shows similar, or even better penetration of slag within said composition, thereby demonstrating the ability of the composition according to examples E1, E2 and E4 to equally, or even better withstand slag corrosion.

It was also observed that all examples 1, 2, and 4 (E1, E2, E4) show excellent results in terms of circular economy, due to the use of second fractions originating from recycled materials, as compared to comparative example 1 and 2.

Likewise, it was observed that all examples 1, 2 and 4 (E1, E2, E4) show excellent results in terms of quarry life.

Hence it was demonstrated that, besides having excellent resistance against slag corrosion, the composition (C) according to examples E1, E2, and E4 take part in circular economy as well as shows excellent quarry life.

Corrosion test

The corrosion and shrinkage of compositions (C) according to examples 1, 2 and 4 (E1, E2, E4) and comparative examples 1 - 2 were evaluated.

Drillings were performed into high purity magnesia bricks (98 % MgO) with 58 mm diameter and 70-74 mm depths. Each of the composition (C) according to examples 1, 2 and 4 (E1, E2, E4) and comparative example 1-2 were hand-rammed into one of the drillings. The hand-ramming was performed without any mechanical densification despite the pressure applied by hand to represent the way the material is applied to the furnace. An EAF slag, having the same composition as detailed herein-above in the thermodynamic calculations, was first ground to a fine powder, before introducing 40-50 g into the cavity of the rammed drillings. Each of the obtained samples were fired in a gas kiln with a heating rate of 5-8 °C/min up to 1700 °C. This temperature was held for 6 h. After the test, each of the samples were sectioned, and analysed.

The quantification of the shrinkage of composition (C) according to 1, 2 and 4 (E1, E2, E4) and comparative examples 1 and 2 was measured on the entire height (side walls) of the fired drillings, and compared to the initial height (side walls) of the drillings. The lowest point of the sample bottom was chosen to measure the difference compared to the unfired samples, which was attributed to the shrinkage and slag corrosion simultaneously. The shrinkage is assumed to occur homogenously in the samples.

The difference of shrinkage and calculated corrosion of applied compositions (C) according to example 1, 2 and 4 (E1, E2, E4) are compared to comparative example 1.

Furthermore, it was showed that the composition (C) according to example 4 (E4) shows excellent corrosion resistance properties compared to comparative example 2 (calculated relative corrosion = -2.1 %).

It was therefore demonstrated that the composition (C) according to example E4 shows excellent corrosion resistance, when said composition (C) is used as hot-repair material.

Examples 6-7 and comparative example CE4

A. Preparation of the composition (C) of examples 6-7 and comparative example CE4

Compositions (C) according to examples 6-7 (E6-E7) and comparative examples 4 (CE4) were prepared by mixing the first fraction 1C with the second fraction 2D and/or 2E and/or the comparative second fraction (fraction 2CE) in amounts as summarized in Table 2. All contents are given in wt.% unless stated otherwise.

B. Properties of the composition (C) of examples 6-7 and comparative example 4

Measurement of bulk and tapped density/Hausner ratio

The calculation of the bulk and tapped density of the composition (C), and its Hausner ratio, according to examples E6-E7 and comparative example CE4 was performed with a Granupack™ tapped density analyser apparatus from Granutools™. Between 30 and 40 cm^3 of the composition (C) was filled in a cylinder. The bulk density, corresponding to the density of the composition at the beginning of the experiment was recorded. A light hollow cylinder is placed on the top of the composition (C) bed to keep the powder/air interface flat during the compaction process. The tube containing the powder sample rose up to a fixed height of ΔZ and performs free falls. The height h of the powder bed is measured automatically after each tap. From the height h, the volume V of the pile is computed. As the powder mass m is known, the density ρ is evaluated and plotted after each tap. The density is the ratio between the mass m and the powder bed volume V. The hollow cylinder was tapped 1250 times and the density evolution was recorded. The tapped density values, corresponds to the density obtained after the cylinder was tapped 1250 times.

The difference between the tapped density and the bulk density Δd was calculated and is summarized in Table 2. The Hausner Ratio is thereby obtained by dividing the tapped density by the bulk density and is summarized in Table 2.

Flowability test

A flowability test of the composition (C) according to examples E6-E7 and comparative example CE4 was performed with a Granudrum™ powder rheometer apparatus from Granutools™. The composition (C) was filled in a transparent double window drum cylinder. Said cylinder is rotated at velocity of 8 rpm and pictures of the composition (C) were taken. The pictures were analysed and the cohesive index, which only relates to the cohesive forces between the particles, was calculated. The cohesive indexes of composition (C) are summarized in Table 2.

The cohesive index, being only related to the cohesive forces between the particles is an indicator of the flowability of the composition (C). For example, a low cohesive index indicates that the composition (C) is flowable. To the contrary, a high cohesive index indicates that the composition (C) is a cohesive powder, thereby a powder having a low flowability.

Table 2

		Composition (C)		
		CE4	**E6**	**E7**
Particulate mixture				
Fraction 1C (wt.%)		75	75	75
%CaO =53.7% % MgO = 37.0 % %Fe_2O_3 = 7.5%	d_{10} = 400 µm d_{50} = 1730µm d_{90} = 3000 µm			
Fraction 2D (wt.%)		0	0	25
%CaO = 24.7% % MgO = 66.6%	d_{10} = 26 µm d_{50} = 462 µm d_{10} = 1091 µm			
Fraction 2E (wt.%)		0	25	0
%CaO = 42.9% % MgO = 30.0%	d_{10} = 27 µm d_{50} = 160 µm d_{90} =856 µm			
Fraction 2CE (wt.%)		25	0	0
%CaO = 29.8% % MgO = 20.9%	d_{10} = 5 µm d_{50} = 37 µm d_{90} = 442 µm			
Chemical composition of the particulate mixture				
% (SiO_2) + % Al_2O_3		3.5	2.3	2.3
% MgO		33.0	35.3	44.4
Ratio d_{50} second fraction / d_{50} first fraction		0.02	0.092	0.267
Δd		0.43	0.29	0.30
Hausner ratio		1.31	1.19	1.19
Cohesive index at 8 rpm		not measurable	31.2	32.5

It was shown that compositions (C) according to examples E6-E7 all shows good Hausner ratio and cohesive index, indicative of an good flowability of the composition (C).

However, the cohesive index at 8rpm of composition (C) according to comparative example CE4, could not be measured, as the composition (C) according to comparative example 4 showed a high cohesive index which was out of the range of measurement of the apparatus.

Industrial test of comparative example CE4:

The composition (C) according to example CE4 is mixed with a oiling compound at a ratio composition (C)/oiling compound of 96/4. The mixture (M) thereby obtained was charged into a hopper comprised in a fettling machine. The mixture (M) was fettled onto refractory lining (L), wherein the refractory lining (L) was at a temperature of between 1100 and 1600°C. It was observed that the mixture (M) was applied unevenly on the refractory lining (L). Furthermore, it was observed that during the operation, clogging of the hopper comprised in the fettling machine occurred. Thus, the test thus was interrupted and could not be terminated

CLAIMS

1. A composition [composition (C), herein-after] suitable for hot repairing refractories, comprising a particulate mixture consisting of at least one first fraction consisting of first particles having a first chemical composition and at least one second fraction consisting of second particles having a second chemical composition, wherein the first fraction has a first d_{50} particle size value between 0.5 – 10 mm and the at least one second fraction has a second d_{50} particle size value, wherein the ratio between the second d_{50} particle size value of the second fraction and the first d_{50} particle size value of the first fraction is from 0.05 to 0.95, and
wherein the first chemical composition, relative to the total weight of the first chemical composition comprises:

- from 20.0 wt.% to 45.0 wt.% of at least one first magnesium oxide compound, as expressed in wt.% of MgO;
- from 40.0 wt.% to 60.0 wt.% of at least one calcium oxide compound, as expressed in wt.% of CaO;
- from 5.0 wt.% to 12.0 wt.% of at least one iron oxide compound, as expressed in wt.% of Fe_2O_3, and

wherein the second chemical composition comprises at least one second magnesium oxide compound, wherein the total content of the first and the second magnesium oxide compounds in the particulate mixture, relative to the total weight of the particulate mixture is from 30.0 to 80.0 wt.%, as expressed in wt.% of MgO.

2. The composition (C) according to claim 1, wherein the first fraction has a first d_{50} particle size value between 0.6 – 8.0 mm, more preferably between 0.6 – 6.0 mm, even more preferably between 0.8 – 6.0 mm, even more preferably between 0.9 - 4.0 mm, even more preferably between 1.0 – 3.0 mm, even more preferably between 1.0 – 2.0 mm.

3. The composition (C) according to any one of claims 1 or 2, wherein the first fraction, has a first d_{10} particle size value equal to or higher than 100.0 µm, a first d_{50} particle size value of between 0.6 – 8.0 mm, and a first d_{90} particle size value equal to or less than 10.0 mm, preferably a first d_{10} particle size value equal to or higher

than 200.0 µm a first d_{50} particle size value of between 0.8-6.0 mm, and a first d_{90} particle size value equal to or less than 8.0 mm, more preferably a first d_{10} particle size value equal to or higher than 300.0 µm a first d_{50} particle size value of between 1.0-2.0 mm, and a first d_{90} particle size value equal to or less than 4.0 mm.

4. The composition (C) according to any one of claims 1 to 3, wherein the amount of the first magnesium oxide, relative to the total weight of the first chemical composition, is equal to or more than 22.0 wt.%, more preferably equal to or more than 25.0 wt.%, even more preferably equal to or more than 30.0 wt.%, as expressed in wt.% of MgO.

5. The composition (C) according to any one of claims 1 to 4, wherein the amount of the iron oxide compound in the first chemical composition, as detailed above, relative to the total weight of the first chemical composition, is from 5.5 to 11.5 wt.%, preferably from 6.0 to 11.0 wt.%, more preferably from 6.5 to 10.5 wt.%, as expressed in wt.% of Fe_2O_3.

6. The composition (C) according to any one of claims 1 to 5, wherein the second fraction has a second d_{50} particle size value, wherein the ratio between the second d_{50} particle size value of the second fraction and the first d_{50} particle size value of the first fraction is from is from 0.08 to 0.85, or from 0.10 to 0.75, or from 0.10 to 0.65, or from 0.10 to 0.50, or from 0.10 to 0.45, or from 0.10 to 0.40, or from 0.10 to 0.30.

7. The composition (C) according to any one of claims 1 to 6, wherein the second fraction has a second d_{50} particle size value of between 50.0 µm and 1000.0 µm, or of between 60.0 µm and 750.0 µm, or of between 70.0 µm and 600.0 µm.

8. The composition (C) according to any one of claims 1 to 7, wherein the total content of the first and the second magnesium oxide compounds, relative to the total weight of the particulate mixture, is from 32.0 to 75.0 wt.%, preferably from 34.0 to 70.0 wt.%, more preferably from 37.0 to 65.0 wt.%, even more preferably from 37.0 to 60.0 wt.%, even more preferably from 37.0 to 55.0 wt.%, even more preferably from

37.0 to 50.0 wt.%, even more preferably from 37.0 to 48.0 wt.%, as expressed in wt.% of MgO.

9. The composition (C) according to any one of claims 1 to 8, wherein the amount of the second magnesium oxide compound in the second chemical composition, relative to the total weight of the second chemical composition is from 20.0 to 80.0 wt.%, preferably from 30.0 to 75.0 wt.%, more preferably from 40.0 to 70.0 wt.%, even more preferably from 60.0 to 70.0 wt.%, as expressed in wt.% of MgO.

10. The composition (C) according to any one of claims 1 to 9, wherein the second fraction originates from recycled materials selected from the group consisting of recycled magnesia and doloma refractory bricks.

11. The composition (C) according to any one of claims 1 to 10, wherein the first fraction and the second fraction, are present in a weight ratio first fraction/second fraction of between 90/10 to 50/50, preferably of between 85/15 to 55/45, more preferably of between 75/25 to 60/40.

12. The composition (C) according to any one of claims 1 to 11, wherein the particulate mixture is substantially free of aluminum oxide compound and silicon oxide compound.

13. The composition (C) according to any one of claims 1 to 12, wherein the composition (C) further comprises at least one oiling compound selected from mineral oil and petroleum jelly.

14. A composition (C) suitable for hot repairing refractories, comprising a particulate mixture consisting of at least one first fraction consisting of first particles and at least one second fraction consisting of second particles, wherein the first fraction has a first d_{50} particle size value between 0.5 – 10 mm and the second fraction has a second d_{50} particle size value wherein the ratio between the second d_{50} particle

size value of the second fraction and the first d_{50} particle size value of the first fraction is from 0.05 to 0.95, and

wherein said first fraction has a second chemical composition according to any one of claims 1-13,

and wherein said second fraction has a first chemical composition according to any one of claims 1-13.

15. A method for the manufacturing of the composition (C) according to any one of claim 1 to 14, wherein the method comprises the step of providing a particulate mixture according to any one of claims 1 to 13, by admixing of:

- from 48.0 to 87.0 wt.%, preferably from 58.0 to 72.0 wt.% of the at least one first fraction;
- from 9.0 to 48.0 wt.%, preferably from 24.0 to 38.0 wt.% of the at least one second fraction; and
- optionally at least one oiling compound.

16. The method according to claim 15, wherein the at least one first fraction, is first mixed with the at least one second fraction, thereby forming the particulate mixture, said particulate mixture is then further optionally mixed with the at least one oiling compound.

17. A method for hot-repairing a refractory lining [refractory lining (L) hereinafter], wherein the refractory lining (L) is hot-repaired with the composition (C) according to any one of claims 1 to 14, wherein the composition (C) is applied to the refractory lining (L) by fettling, spraying, or gunning the composition (C), by using conventional equipment such as a dedicated fettling machines.

18. The method according to claim 17, wherein the composition (C) is applied on the refractory lining (L) having a temperature of at least 1200 °C, or at least 1300 °C, or at least 1400 °C.

19. A sintered hot repair-material obtained by the method for hot-repairing a refractory lining (L) according to any of claims 17 or 18.

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2022/067280

A. CLASSIFICATION OF SUBJECT MATTER
INV. C04B35/043 C04B35/057 C04B35/622 C04B35/66 C04B41/52
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C04B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 2 943 240 A (MARTINET JACQUES R) 28 June 1960 (1960-06-28) cited in the application column 7, line 36 - column 8, line 3 -----	1-19
A	WO 2010/024497 A1 (WONJIN WORLDWIDE CO LTD [KR]; SON DAL HO [KR]) 4 March 2010 (2010-03-04) claims -----	1-19

☐ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

* Special categories of cited documents :

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"P" document published prior to the international filing date but later than the priority date claimed

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Date of the actual completion of the international search 1 September 2022	Date of mailing of the international search report 09/09/2022
Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Authorized officer Rosenberger, Jürgen

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/EP2022/067280

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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