(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) Publication number: **0 074 715**
A1

(12) # EUROPEAN PATENT APPLICATION

(21) Application number: **82304192.6**

(51) Int. Cl.³: **C 21 D 8/12**

(22) Date of filing: **09.08.82**

(30) Priority: **24.08.81 US 295591**

(43) Date of publication of application:
23.03.83 Bulletin 83/12

(84) Designated Contracting States:
BE DE FR GB IT SE

(71) Applicant: **ALLEGHENY LUDLUM STEEL CORPORATION**
Oliver Building 2000 Oliver Plaza
Pittsburgh Pennsylvania 15222(US)

(72) Inventor: **Shilling, Jack Walther**
1308 Treeline Drive
Murrysville Pennsylvania 15660(US)

(72) Inventor: **Datta, Amitava**
5, Pembrooke Place
Morris Township New Jersey 07960(US)

(74) Representative: **Sheader, Brian N. et al,**
ERIC POTTER & CLARKSON 5 Market Way Broad Street
Reading Berkshire, RG1 2BN(GB)

(54) **Method for producing oriented silicon steel having improved magnetic properties.**

(57) An improvement in the manufacture of oriented silicon steel to improve the magnetic properties, and specifically to achieve improved core loss; the improvement comprising removing as by acid pickling at least 3.3 μm from each surface of said steel and normalizing said steel to effect decarburization prior to final texture annealing.

EP 0 074 715 A1

METHOD FOR PRODUCING ORIENTED SILICON STEEL HAVING IMPROVED MAGNETIC PROPERTIES

The present invention relates to a method for producing oriented silicon steel having improved magnetic properties and particularly improved core loss.

Oriented silicon steel in the form of sheets is known for use in various electrical applications, including the manufacture of transformer cores. The steel is produced by hot-rolling followed by cold-rolling with or without intermediate annealing. Normalizing treatments are then conducted during which both decarburization and recrystallization are achieved. The steel is then conventionally coated and texture annealed. With oriented silicon steel after final texture annealing the alloy is characterized by a secondary recrystallization texture in the (110) (001) position, which is termed the cube-on-edge orientation. This alloy in sheet form has a single direction of easy magnetization in the direction of rolling. In applications for this material and specifically when used in the manufacture of transformer cores the material desirably has reduced core loss, because the consumption of electrical energy decreases as core loss decreases. Reduced

core loss may be promoted by achieving improved cube-on-edge orientation or grain structure, which in turn results in improved magnetic properties, specifically improved core loss.

It is accordingly an object of the present invention to provide a method whereby oriented silicon steel may be provided with an improved orientation with regard to the secondary grain or crystal structure after texture annealing, which achieves reduced core loss.

The present invention provides a method for producing oriented silicon steel characterized by improved core loss, including the steps of hot-rolling, cold-rolling with intermediate annealing, normalizing and final texture annealing, wherein at least 3.3μm is removed from each surface of said steel after cold-rolling and prior to normalizing said steel.

The invention will be more particularly described with reference to the accompanying drawings, in which:-

Figure 1 is a schematic representation of specific examples of the practice of the present invention;

Figure 2 is a curve showing the permeability achieved with respect to the specific examples set forth in Figure 1; and

Figure 3 is a curve showing the core loss determinations with respect to the specific examples of Figure 1.

In accordance with the invention it was determined better oriented secondary grains are produced in the interior of a strip as opposed to the surface portions thereof. In accordance with the practice of the invention it was determined that, and as will be shown by specific examples hereinafter, if

oriented silicon steel is subjected to an operation such as acid pickling to remove at least 3.3 μm from each surface of the steel after cold-rolling and prior to normalizing and final texture annealing the surface portion thereof will be characterized by an improved secondary recrystallization or grain orientation which is the substantial equivalent of that typically occurring in the interior of the strip. This removal operation is performed by acid pickling and preferably by the use of a hydrochloric acid solution.

By way of a specific example to demonstrate the invention samples of oriented silicon steels of the following compositions were employed:-

Heat	C	Mn	S	Si	Cu	B	Nz
494	030	034	020	3.12	.35	.0011	.0043
495	043	035	020	3.24	.34	.0004	.0049

Samples in the form of strips of the above compositions were processed to determine the effect of surface removal brought about by pickling on the final magnetic properties after texture annealing. The processing sequences for these samples are set forth in Figure 1. As may be seen from this Figure 1 samples of the steel were subject to pickling before, after and between final normalizing steps. In Figure 1 the symbols N, Q and P indicate the following treatments:

N = Normal final normalizing cycle, 802°C (1475°F), 152.4 (6 in)/min., in $80N_2/20H_2$ at 10°C (50°F) d.p.

Q = Quick heat to 982°C (1800°F) for 1 min. in $80N_2/20H_2$ at 10°C (50°F)d.p.

P = Pickle in 50% HCl, removing about ½ mil. from each side of strip.

Prior to texture annealing all the sample strips were coated with MgO + .75%B and thereafter final texture annealed in H_2 at 1177°C (2150°F). The magnetic properties of the samples after final texture annealing with respect to permeability and core loss are set forth in Figures 2 and 3, respectively. The number after each data point in Figures 2 and 3 represents the calculated gauge of the strips. By way of comparison with conventionally processed material not subjected to pickling in accordance with the examples set forth in Figure 1, the permeability and core loss data for conventionally processed materials are set forth in Table I.. The material of Table I is of the same composition as that subjected to the testing as reported in Figure 1 and likewise was similarly coated after a similar normalizing treatment and prior to texture annealing.

TABLE I

Heat #	Final Norm. Cycle	Coating	10H	WPP 17KB
148494	802°C (1475°F), 152.4mm (6") min., $80N_2$ /$20H_2$ at 15°C (60°F) d.p.	MagS + .8%B	1890	.711
148495	802°C (1475°F), 152.4mm (6") min., $80N_2/20H_2$ at 15°C (60°F) d.p.	MagS + .8%B	1897	.681

From the magnetic property data presented in Figures 2 and 3, when compared with the Table I data for conventionally processed material, it is evident that pickling in accordance with the invention prior to final normalizing results in an appreciable improvement in magnetic properties after texture annealing. More specifically, the treatment for the samples

as set forth in Figure 1 designated at P + Q treatment yields the lowest core loss; whereas, the sample subjected to the N + P treatment produces the highest core loss. The N + P treatment samples with respect to core loss approximate the core loss values achieved with conventional processing, absent a pickling treatment, as may been seen form the Table I data.

CLAIMS:

1. A method for producing oriented silicon steel characterised by improved core loss, including the steps of hot-rolling, cold-rolling with intermediate annealing, normalizing and final texture annealing, characterised in that at least 3.3μm is removed from each surface of said steel after cold-rolling and prior to normalizing said steel.

2. A method according to claim 1, characterised in that at least 3.3μm is removed from each surface of said steel by acid pickling.

3. A method according to claim 2, characterised in that said pickling is performed by the use of an HCl solution.

0074715

Fig. 1.

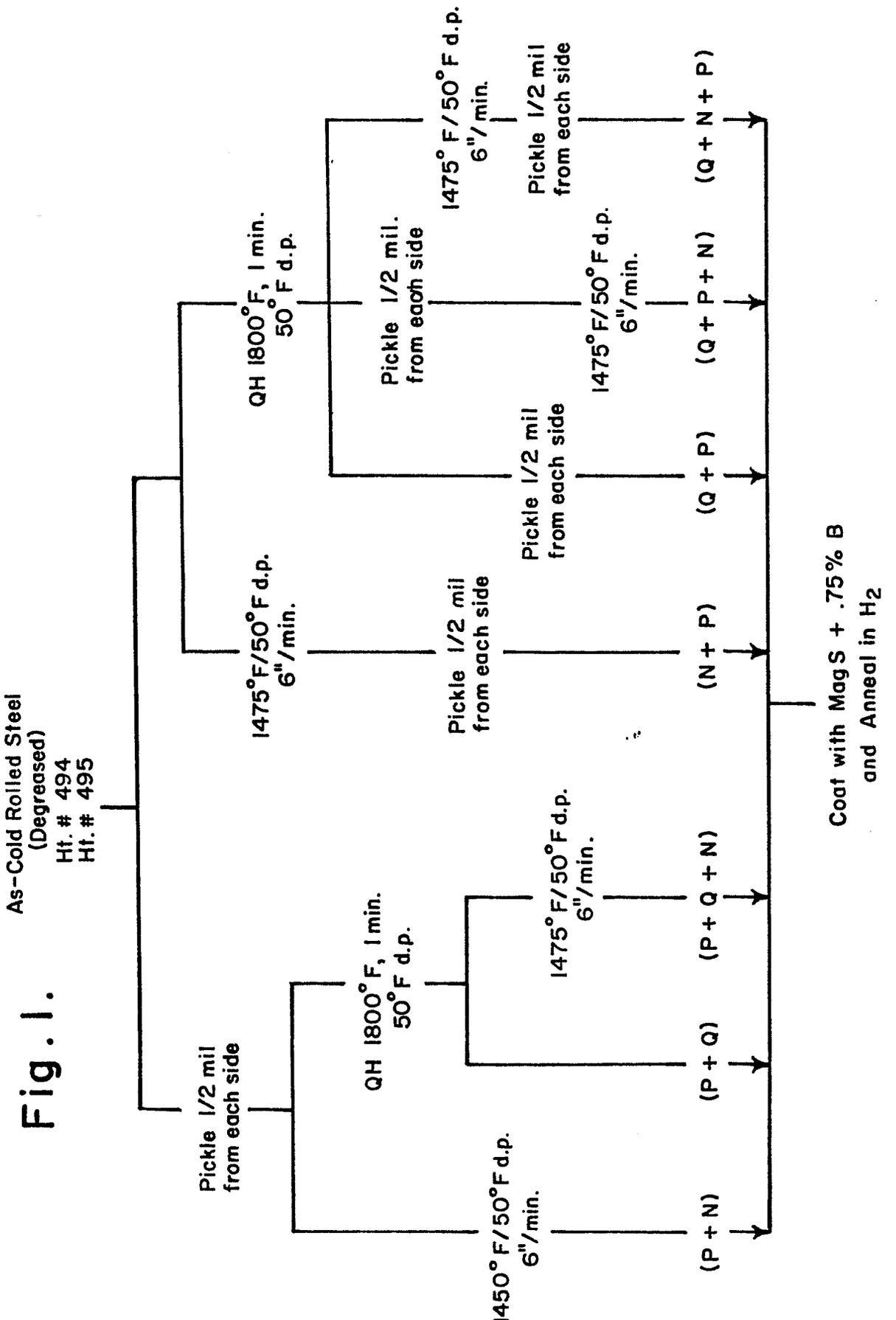


Fig. 2.

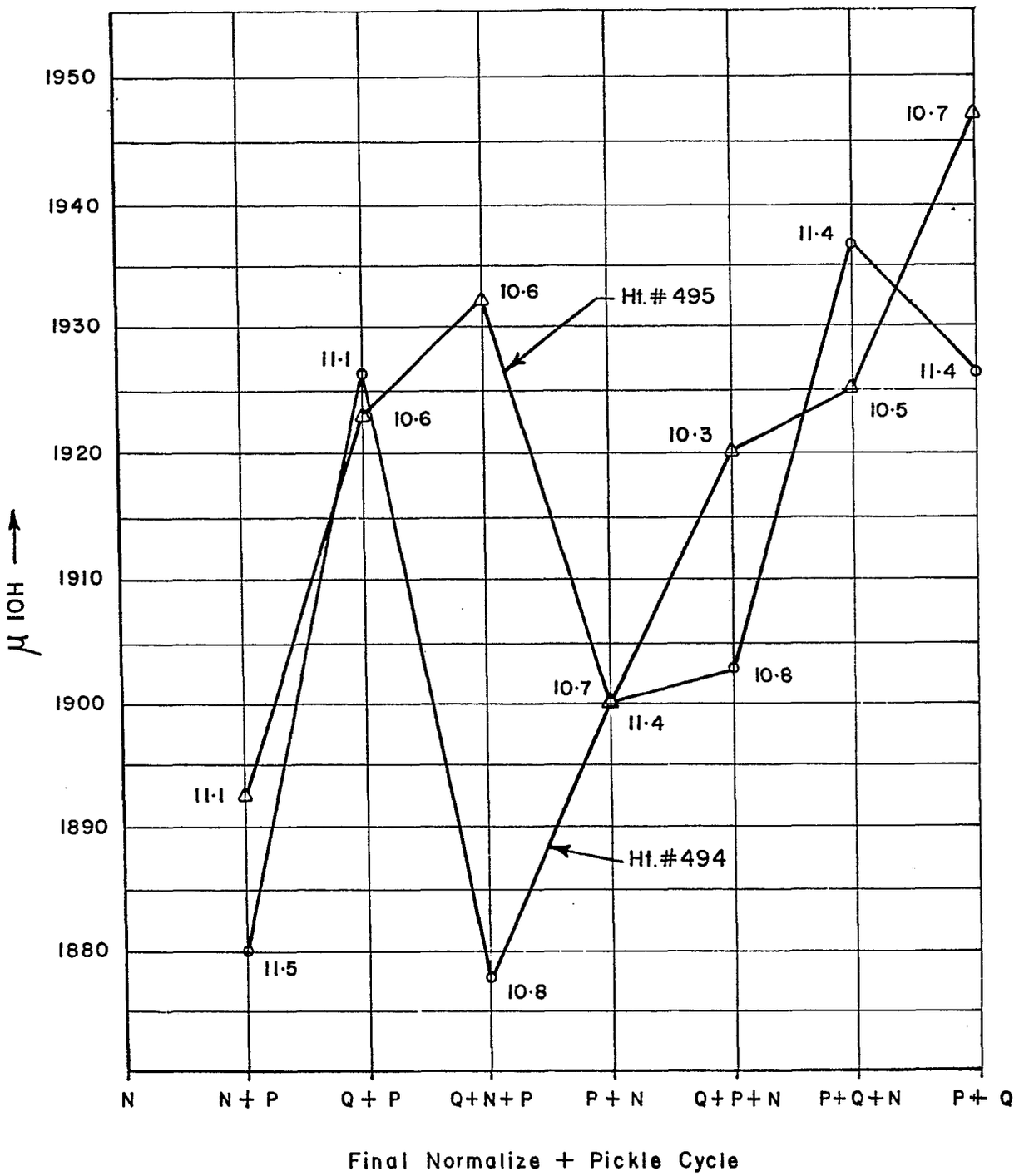
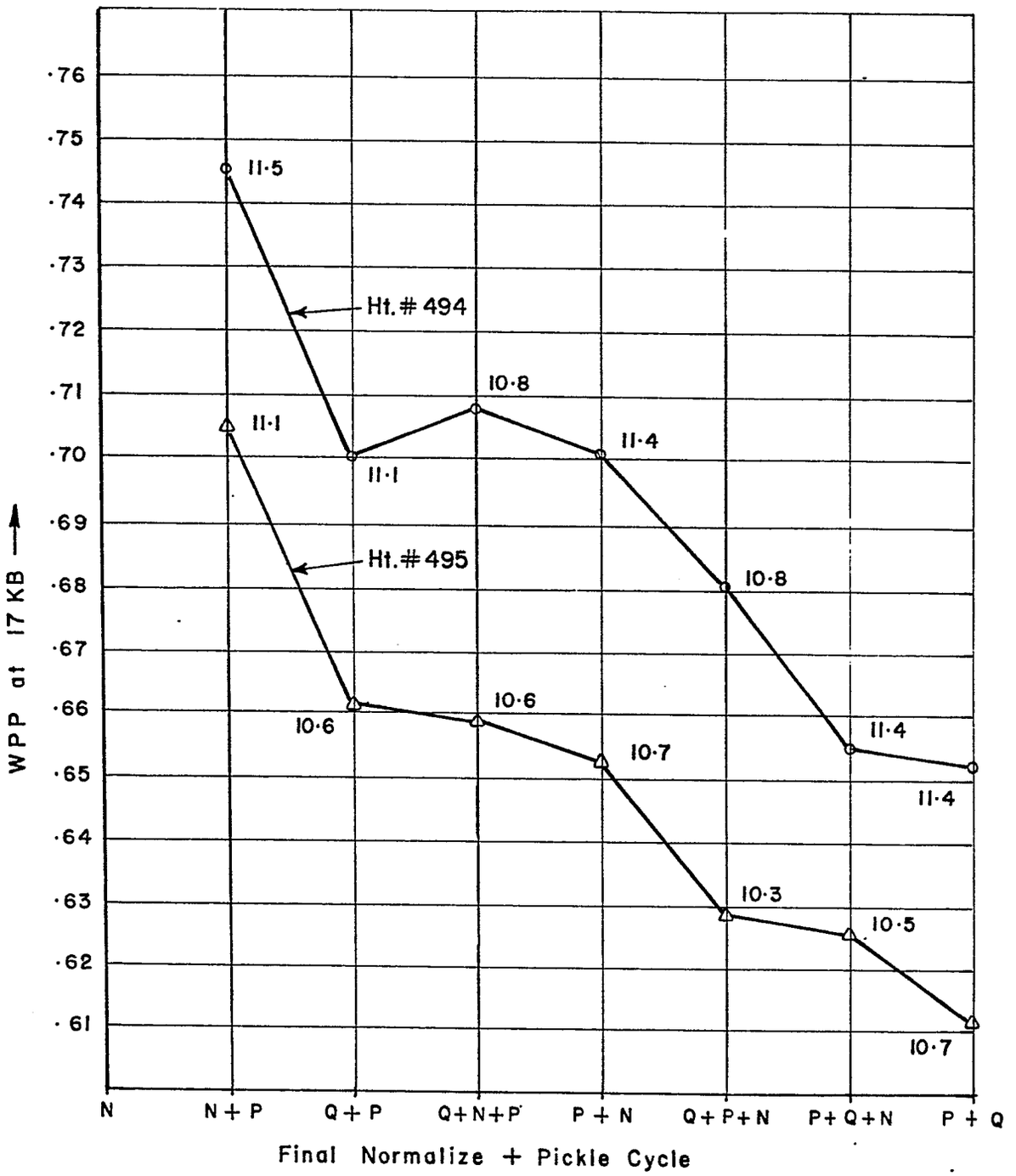

1950
1940
1930
1920
1910
1900
1890
1880
μ IOH
10·7
11·4
10·6
Ht.# 495
11·1
11·4
10·6
10·3
10·5
10·8
10·7
11·4
11·1
Ht.#494
11·5
10·8
N
N + P
Q + P
Q+N+P
P + N
Q+P+N
P+Q+N
P+ Q
Final Normalize + Pickle Cycle

Fig.3.


·76
·75
·74
·73
·72
·71
·70
·69
·68
·67
·66
·65
·64
·63
·62
·61
WPP at 17 KB
11·5
Ht.# 494
11·1
10·8
11·4
10·8
11·4
Ht.# 495
11·1
10·6
10·6
10·7
10·3
10·5
10·7
N
N + P
Q + P
Q+N+P
P + N
Q+P+N
P+Q+N
P + Q
Final Normalize + Pickle Cycle

European Patent Office

EUROPEAN SEARCH REPORT

0074715
Application number

EP 82 30 4192.6

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. 3)
Y	US - A - 3 105 781 (J.L. WALTER) * column 3 * --	1	C 21 D 8/12
Y	US - A - 4 213 804 (A. DATTA) * col. 1 to 4 * --	1	
A	US - A - 3 090 711 (D.M. KOHLER) --		TECHNICAL FIELDS SEARCHED (Int.Cl. 3)
A	US - A - 3 347 718 (V.W. CARPENTER et al.) --		C 21 D 8/12
A	US - A - 3 932 236 (T. WADA et al.) --		
A	US - A - 4 054 471 (A. DATTA) --		
A	DE - B - 1 111 225 (WESTINGHOUSE ELECTRIC) --		
A	DE - B - 1 191 399 (WESTINGHOUSE ELECTRIC) ----		

CATEGORY OF CITED DOCUMENTS

X: particularly relevant if taken alone
Y: particularly relevant if combined with another document of the same category
A: technological background
O: non-written disclosure
P: intermediate document
T: theory or principle underlying the invention
E: earlier patent document, but published on, or after the filing date
D: document cited in the application
L: document cited for other reasons

&: member of the same patent family, corresponding document

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
Berlin	25-11-1982	SUTOR

EPO Form 1503.1 06.78